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EVALUATION OF POLYIMIDE/GLASS FIBER COMPOSITE FOR CONSTRUCTION OF LIGHT WEIGHT PRESSURE VESSELS FOR CRYOGENCI PROPELLANTS

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16. Abstract

The application of polyimide resin as a matrix for glass filament-wound thin metal-lined pressure vessels was studied over a temperature range of -320 to 600°F. Keramid 601 polyimide was found to perform quite well over the entire range of temperature. Hoop stress values of 425 ksi were determined at 75°F which is equivalent to epoxy resin in similar structures. At -320 and 600°F, 125 and 80% of this strength was retained. Thermal ageing at 500°F for up to 50 hours was studied with severe reduction in strength, but there is evidence that this reduction could be improved. Another polyimide resin studied was P10PA which was found to have processing characteristics inappropriate for filament-winding. NOL ring tensile and shear data was determined from both resins with S-glass. Pressure vessel design, fabrication and test procedures are described in detail.

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FORWARD

This report is submitted by Structural Composites Industries, Incorporated in fulfillment of the contract. It covers all work on the program, which was conducted from June 1971 to September 1972 for the Lewis Research Center under Mr. Raymond Lark's technical direction. Mr. Ira Petker was SCI's Program Manager and Mr. Masaru Segimoto was the Project Engineer. Design analysis was accomplished by Mr. Robert E. Landes and vessel testing by Mr. Kenneth Hansen. Consultation with Mr. Edgar E. Morris on various matters was helful throughout the program.

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EVALUATION OF POLYIMIDE/GLASS FIBER COMPOSITES FOR CONSTRUCTION OF LIGHT WEIGHT PRESSURE VESSELS FOR CRYOGENIC PROPELLANTS

by M. Segimoto and I. Petker

Structural Composites Industries, Inc.

SUMMARY

The objective of this program was to determine the feasibility of using polyimide resin as a matrix for metal-lined filament-wound glass composite pressure vessels. Two matrices were evaluated based on their improved processability over classical polyimides. These were Gemon-L (General Electric proprietary material, withdrawn from the market, and succeeded by Rhodia Corporation's Keramid 601) and P10PA, a resin developed under NASA/Lewis Research Center sponsorship, which cures by pyrolitic decomposition and addition.

The program consisted of the preparation and testing of unidirectional composites with S-glass to define material and processing parameters, the design of a 4-inch-diameter x 6-inch-long closed-end pressure vessel with a thin, 0.005 - 0.007-inch-thick stainless steel liner, the procurement of liners, and the preparation and test of the pressure vessels between -320 and 600°F, after thermal ageing and after thermal cycling.

The ability to filament wind high quality composites with Gemon-L had been proven and a basic process was available by SCI prior to the program. Therefore, it was sufficient to restrict the material and processing study for this resin towards optimizing certain parameters for a filament winding process. Similar background did not exist for P10PA and an exploratory study was conducted to identify the feasibility of this resin for

filament winding. The results of this study were not sufficiently encouraging to incorporate it into vessels on this program, although it should be understood that the processing study of P10PA was very limited and narrow, and with additional effort it might qualify for filament winding.

All vessel work was conducted with Gemon-L. Hoop stress values of 425 ksi were determined at 75°F which is equivalent to epoxy matrix in similar structures. At 600°F, 80% of this strength was retained initially. After 100 hours, 80 % of the original strength was retained and after 500 hours, 50 % was retained. As noted in the text, there is evidence that the thermal strength retention of this resin could be improved.

I. INTRODUCTION

Filament-wound composite pressure vessels with metal liners have been under development for about ten years. These vessels exploit the uniquely high specific tensile strengths of structural fibers and the resistance to chemical attack and very low vapor transmission of certain metals. From the perspective of the metal liner, two general categories of vessels have evolved: (a) those in which the liner is relatively thick so that it can withstand the pressures of the composite overwrap and is designed to share the structural load with the composites, and (b) those in which the liner is relatively thin so that it would buckle unless supported, by adhesive bonding to the inside wall of the vessel, and serves only as a barrier against molecular diffusion and chemical attach of the contained fluids.

For cryogenic service metal liners also provide both the extensibility and leak-tightness which cannot be achieved with polymeric materials. The ability of thin metal-lined tanks to achieve substantial weight savings over homogeneous metal tanks has been well documented (References 1, 2, and 3). This work was confined to a temperature region between ambient (75°F or 297°K) and liquid hydrogen (-423°F or 20°K). The ability of these tanks to perform at higher temperatures is limited by the matrix used in the composite. Only epoxy resin with moderate ability to perform at temperatures above ambient have had the necessary combination of structural and processing characteristics to be considered for these pressure vessels. However, recent improvement in the processability of polyimide resins, which retain significant strength to temperatures of 600°F (or 558°K) and also are useful cryogenically

give rise to the potential development of a metal-lined composite tank capable of -423°F to 600°F. The objective of this program was to demonstrate the feasibility of such a tank consisting of a thin stainless steel liner over-wrapped with glass fibers and a polyimide matrix.

II. POLYIMIDE MATRIX EVALUATION

Two polyimide resins were chosen as candidates for the matrix: Gemon-L[1] and P10PA. Both resins polymerize via addition reaction and are differentiated by the higher thermal stability and higher melt temperature of P10PA. The former attribute is obviously desirable, whereas the latter attribute imposes greater difficulties in processing. At the beginning of this program, P10PA was still a very new material with its appropriateness to filament winding not yet established. Thus an early investigation was an attempt to establish a processing method for filament winding P10PA. Conversely, the feasibility to filament wind with Gemon-L had already been established by SCI and a parametric process study to optimize its processing could be initiated without preliminary study.

A. P10PA EVALUATION

1. Preliminary Study

The primary processability characteristic of a matrix which is desirable for filament winding preimpregnated tapes is the ability to achieve, during the winding process, a physical state of such plasticity that layers of fiber will tack to the mandrel and progressively to succeeding layers of prepreg. If the viscosity of the matrix during winding is properly controlled, there will be local flow which assists in thorough wetting of fibers and minimization of occluded air. Although a pressure vessel is less dependent upon low void content for its structural performance than most other kinds of composite structures, voids will reduce thermal stability and, if sufficiently high, static performance. In this study an attempt was made to determine if P10PA could be processed in this manner.

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(1) The polyimide resin, marketed as prepreg under the trade name "Gemon-L" by General Electric Company is now supplied as a neat resin by the Rhodia Corporation under the trade name "Keramid 601".

P10PA was prepared in the form of a varnish in dimethyl formadide (DMF) by TRW Systems Group, Redondo Beach, California. This is one of a series of polyimide materials developed under NASA/Lewis sponship based on blocked, low molecular weight polyamic acids which polymerize via pryolytic decomposition followed by addition polymerization after imidication. These resins are characteristically solid in both the amide acid and imidized state, thus requiring solvents for impregnation. PI3N has been commercialized by Geigy-Ciba and used in compression molding processes with substantial success. Attempts to filament wind PI3N have not been successful, primarily because of the short gel time and high viscosity of the imidized melt. P10PA was designed to extend the gel time of the melt and reduce its viscosity.

Two batches of P10PA polyimide resin was received for evaluation in this program. Resin solutions were characterized for solids content. Two resin samples from each container tested were weighed in tared aluminum cups and residual solids were determined after exposure to heat at 500°F for thirty minutes. The solids content of the two resin batches were 35.3 and 36.7 weight percent, which is equivalent to the solids content of the commercially available PI3N polyimide resin. Results of the resin characterization tests for the two batches are summarized in Table 1.

A cursory evaluation to define thermal conditioning requirements was conducted with the P10PA resin impregnated with Style 181 glass fabric. The impregnated fabric was cut in nine equal pieces and was treated each to a different time-temperature condition of 15, 30, and 60 minutes at 180, 200, and 250°F. The treated prepreg materials were examined for volatile contents, tack, resin brittleness, and flow and wetting characteristics of the material on a 250°F platen. The high tack and wet condition of the 180°F materials indicated that this temperature was insufficient for thermal conditioning. The prepregs treated at 200°F for

60 minutes and at 250°F for 15 minutes appeared to have advanced the material to a suitable level. The volatile contents of the materials were 25.2 and 23.3 weight percent, respectively, based on the prepreg resin weight. Gravimetric data and qualitative processability comments are given in Table II.

Nineteen batches of prepreg roving were prepared by a drum winding process. Two preliminary runs were made to establish the prepreg resin-content control and to determine the thermal conditioning requirements of prepregs for filament winding applications. One batch of prepreg roving, S/N 1, was fabricated to a target resin content of 25 weight percent and the other, S/N 2, to 32 weight percent. Both of these target values were achieved satisfactorily. The prepregs were treated at 200 and 230°F for 60 minutes using the information developed earlier from the glass-fabric thermal treatment. Gravimetric data for these and the other prepregs are given in Table III.

NOL rings were wound from the two trial prepregs to determine the heating requirements and processing characteristics. Some traces of resin flow were noted during winding on a steel NOL mandrel heated at 300°F. The postcured rings were generally dark in appearance, but had many light-colored streaks in the interior windings, indicating insufficient resin-flow during winding and cure. Many interlaminar delaminations were clearly visible from the side of the ring. The poor composite integrity was demonstrated by the cracking sound emitted when the rings were flexed by compressing the outer rim. It was apparent from the initial attempt in fabricating the PI0PA NOL rings that a processing study was necessary to develop a fabrication procedure for this resin system.

A processing study was conducted in limited scope with the PI0PA prepreg roving. Processing variables evaluated were winding

tension, devolatilization temperature and time, and cure pressure applied by means of a glass-roving overwrap. Fourteen NOL rings were fabricated using two batches of the 28% resin prepregs which had been treated at 200°F for 45 minutes. The volatile content of the prepregs was 31% by weight based on the prepreg resin weight. This is compared with 23 and 26% volatiles for the two materials used in the previous test. It was noted during winding that a substantial amount of resin flowed out when the steel mandrel was heated initially at 250°F and cooled down to about 210°F at the completion of winding. The wound prepregs were imidized at 250 and 350°F for periods up to 24 hours. Various levels of overwrap cure pressure were then applied and cured at 550°F for 4 hours.

Data for these NOL rings is given in Table IV. They generally had better composite integrity than previously as indicated by a high-pitch sound when tapped with a metallic object. However, it was not realized until the composite resin contents were determined that an excessive resin flow had occurred during the winding. Because of the very low resin content, ranging from 11.9 to 18.7 weight percent, of the various NOL rings, the effects of the processing variables had been obscured to a large extent. The study showed that the resin imidized under the test conditions still has sufficient plasticity to be compacted from the applied pressure during cure. This was indicated by the impressions left on the surface resin from the glass-roving overwrap.

The NOL rings were tested for ring-compression modulus and horizontal shear using segments of the rings. The test results, presented in Table V, show that there are no significant effects on the composite properties from the various processing parameters evaluated. A slight trend in the direction of improved composite properties was noted when the windings were subjected for less time to a given imidization

temperature. The ring flexural modulus ranged from 7.5 to 10.0 x 10^6 psi, except for one ring (9-37) which had an obvious flaw. The modulus of this ring was 5.2 x 10^6 psi. The horizontal shear strength ranged from 4,470 to 6,500 psi, whichis no more than one-half the value expected from this resin system under the condition of sufficient resin in the composite and low void content.

Another experiment was conducted to determine the effectiveness of devolatilizing residual solvents from the wound prepreg material. Particular attention was given during winding to prevent excessive resin flow out, as experienced previously. A prepreg test sample was taken after winding each NOL ring. The wound prepregs were subjected to heat at 250°F for 4, 8, and 16 hours and at 325°F for 4 hours. Test samples were cut subsequently from the imidized prepregs for volatile-content determination. One sample from each ring was split in half and each half section tested individually. The test results in Table V show that a considerable amount of volatile matter still remains in the windings after the thermal treatment. The volatile contents were 17.7, 14.7, and 11.4% by weight of prepreg resin after 4, 8, and 16 hours at 250°F. Even after exposure to heat at 325°F for 4 hours, the volatile content was high at 15.2 weight percent. The samples from the outer half of the winding had less volatiles and higher resin solids than the interior half section. The significance of this study was that it revealed the processing of this material system to be much more difficult than anticipated to obtain high-quality, low-void composites in a straight-forward filament-winding process.

The greatest advantage in curing a resin system such as P10PA would be in the ability to wind with it in its imidized state. The fundamental requirement for a winding resin is that it be capable of being plasticized for low and controlled flow at the time it contacts the

mandrel. Epoxy resin can be formulated to accomplish this at ambient temperature. Other materials, staged properly, might require heat. In this case the winding process is more complicated, but not impractical. The dry nature of this prepreg adds to complications in its use, but this also can be accommodated as long as sufficient plasticity can be achieved during winding. The very high temperatures apparently required for P10PA suggested that such a process was not practical. Therefore, either augmented pressure during cure and a high residual solvent content would be required with the winding conducted before immidization. Under these conditions, the processing difficulties are similar to classical polyimides.

When a filament winding material can be processed as a 100% solids material, the need for augmented during cure is eliminated or is very moderate, the winding tension being sufficient to supply compaction pressure. If high augmented pressure is required, autoclaves or other high pressure sources must be used, which is often impractical and may introduce serious defects into the composite since resin displacement under a condition of augmented pressure generally results in buckled fibers and wrinkles.

Our past efforts to successfully wind P13N in an imidized state have been unsuccessful due to the excessive heat and pressure require. We were not satisfied, as yet however, that a compromise in the material processing might not be possible with P10PA. Toward this end, we continued our efforts towards curing P10PA as a pre-imidized prepreg with occluded solvent, the latter adjusted to give good flow behavior at about 250°F during winding with succeeding process steps directed toward eliminating the volatiles while maintaining satisfactory compaction.

An additional twelve NOL rings were fabricated on standard steel mandrels. Processing variables evaluated included devolatilization time at 250°F, subjection of the windings to vacuum

environment by means of a vacuum bag, and cure pressure applied by means of a glass-roving overwrap and/or vacuum bag. The various processing conditions used in this study are shown in Table VI, and the results of the composite evaluation in Table VII including the gravimetric-analysis data, the ring compressive modulus, and horizontal-shear strengths from segments of the NOL rings.

The data in Table VII shows that it is feasible to fabricate fairly low-void-content composites by a filament-winding process. Void content as low as 2.9 volume percent was measured from an NOL ring which was cured with a 500 psi overwrap pressure. The horizontal shear strength of this ring was disappointingly low at 5.88 ksi, considering the low voids and good over all physical appearance of the ring. This strength value was not any better, and in fact, was inferior to some composites which had much higher measured void content. The void content range was 2.9 to 6.3 volume percent and the shear strength range was 4.5 to 7.4 ksi. The ring flexural modulus ranged from $7.9 - 8.9 \times 10^3$ ksi and resin contents were between 15 and 21 weight percent.

There was no improvement in the composite properties from extended devolatilization time. The composites which had only a 4-hour treatment at $250^{\circ}F$ were generally more consistent with respect to composite properties.

The results obtained from this study were less than optimum. However, these results, the best we could achieve without much more extensive study, were encouraging enough to proceed with the composite cylinder fabrication for the winding parameter study.

2. Winding Parameter Study - P10PA Prepregs

Unidirectional filament wound cylinders were

fabricated to identify optimized material and processing variables with P10PA prepregs. Twelve cylinders were fabricated on plaster mandrels. Plaster was used since this was intended to be the support medium for the pressure vessels and it was considered necessary to simulate the thermal behavior of this material during this stage in the study.

Existing tooling available from previous programs was used to prepare cylindrical plaster mandrels for fabricating composite test specimens. A 5-inch-diameter plaster core was cast initially on a 1-1/2-inch-diameter steel shaft and was built up to 6-inch-diameter by sweeping additional plaster on a rotating mounting fixture. Three slots, 2.5-inch wide by 5.75-inch-diameter, were swept in the mandrel using a template fabricated for this purpose. The slots were incorporated in the mandrels so that three cylinders could be wound and cured simultaneously for each of the three winding tensions to be evaluated for each material type.

The plaster mandrels were dried in an oven at 170 to 190°F for 15 to 17 hours and at 350°F for 24 hours. Fine cracks developed during drying in the first group of mandrels fabricated. Some of the cracks extended to the 5-inch-diameter core. One mandrel was damaged beyond repair when the outer shell broke off during the final machining operation.

Other mandrels were subsequently fabricated using some process modifications to improve the adhesion at the interface between the core and the outer shell. This was accomplished by cutting narrow grooves in the plaster core to provide a means for mechanical interlocking, and the core surface was sealed with shellac prior to applying additional plaster. No surface cracks were visible in these mandrels after drying at 350°F.

The dried plaster mandrels were readily machineable to close tolerance, and the surface was generally smooth. The surface was sealed with several coatings of shellac-acetone solutions. The shellac soaked readily into the plaster and produced a hard surface when allowed to dry in air. Thalco 225 mold release was applied on the shellac-treated surfaces prior to winding. Rezolin 833A, a waxy paste plaster sealer, was evaluated for this application. It was somewhat difficult to apply smoothly on the surface. The sealer had a tendency to lift and peel in splotches. Because of this problem, and satisfactory results obtained with shellac, further evaluations with Rezolin 833A were discontinued.

For winding the mandrel were first heated to 220 to 230°F and heat was applied during winding to maintain the desired level of resin flow and material compaction. The wound prepregs were subjected to heat in an air-circulating oven at 250°F for 8 hours. Two layers of glass-roving overwrap were subsequently applied over the prepreg winding using a constant 8-pound tension to provide an estimated 135 psi pressure during cure. The assembly was oven-cured for 4 hours at 550°F.

The cured cylinders had a uniform surface resin layer that was dark and showed every indication of dense and sound composites. The results, however, were very disappointing. All the cylinders were of very poor quality. It was very difficult to cut the cylinders without incurring many damages and the cut edges were generally fuzzy. Many specimens delaminated and fell apart on cutting the rings in attempting to prepare test samples for gravimetric analysis. Only resin contents were determined from four test samples for each cylinder. Rings which had sufficient composite integrity were tested for ring modulus, horizontal shear, and tensile strength. The rings tested were from the two cylinders (C18 and C21) which were both made with 12-pound winding

tension and low resin content.

The results of the composite evaluation with P10PA prepregs are presented in Table VIII. The data shows essentially no differences in resin content among the various composites made from prepregs with considerable resin-content variation. The ring tensile strengths from the two cylinders were 189.3 and 198.0 ksi which were high considering the poor composite quality of these rings. The composite from cylinder C 18 was so poor that horizontal shear test specimens could not be prepared without delaminations.

The large differences in composite quality obtained between these and earlier rings fabricated may have been due to the different heat-up characteristics of steel mandrels and plaster mandrels. A different and slower heat-up rate occurred during cure with the plaster. A slower composite temperature rise probably resulted in insufficient viscosity reduction during cure. There is not adequate information to permit firm conclusions and other factors not apparent may have been responsible for the lack of composite quality.

Because of these discouraging results and the significantly better behavior of Gemon-L work with P10PA was discontinued.

B. GEMON-L EVALUATION

1. Initial Material Problems

Gemon-L prepreg rovings with four levels of resin content were procured from General Electric. The four levels of resin contents are 35, 30, 25, and 22% by weight for 55, 60, 65, and 70 volume percent fibers, respectively. The Gemon-L prepregs were procured with the resin content slightly higher (as recommended by

General Electric), than the calculated amounts needed in the composites. This was due to the dry and brittle condition of the prepreg resin such that 2 to 3% weight loss could be anticipated from the resin flaking off during the winding operation.

There were indications of difficulties in packaging the prepregs on a standard 3-inch-diameter by 10.8-inch-long cardboard core. The prepregs in 2-pound quantity per material type were received in six spools, one of which contained only one-half pound. Four of the larger spools were not in a useable condition for tensioning the prepreg directly on the package, as required for filament winding. The prepreg materials had either slipped and telescoped out beyond the end of the core or the material had been wound in the as-received condition shown in Figure 1. It was the poorest example of roving packaging that we had ever experienced. The four rolls were rewound onto other cores.

The prepregs were characterized for resin content, volatiles, and weight-per-yard of glass roving. Three samples were taken from each spool and additional samples from Roll 1 and Roll 2 (resin contents of 20 and 24 weight percent, respectively) after rewinding the packages. The test data in Table IX shows the resin contents of the different material variations were within the specified range of $\pm$2% variation from the nominal value. Relatively large variations in volatile content were obtained from the materials tested and, in many cases, they were well below the 2% maximum stipulated in the procurement. The volatile content of this material lot was considerably lower than those determined from prepregs procured previously in another study. The former material had good resin flow and processing characteristics, and the composites made from this material were of high quality with superior mechanical properties.

Trial fabrication of a filament-wound composite was made using the 25% resin material on a plaster mandrel. It was impossible with this material to obtain a uniform "wet" appearance in the winding due primarily to poor resin flow. The mandrel and the prepreg were heated to encourage resin flow during winding, but the appearance of the cured composite indicated lack of sufficient flow during winding. The purpose of this trial run was to establish the fabrication procedures and to make any changes, as required, to the preliminary process specification prepared for fabricating ring test specimens.

The prepreg roving was wound on a 250°F preheated mandrel using 8-pound winding tension and a 0.050-inch/turn lead. The mandrel temperature was maintained at approximately 250°F during winding with a quartz strip heater and a heat gun directed at the roving near the pay-off roller and the mandrel. The cure was at 350°F for two hours under a pressure applied by two layers of glass-roving overwrap in addition to a vacuum bag pressure.

It was apparent from the appearance of the winding that the material had insufficient resin flow under the heating and winding conditions. The winding speed was reduced from a planned 20 to 25-feet/minute to approximately 9-feet/minute in an attempt to obtain a higher degree of resin-flow. Essentially, no resin-flow had taken place during the cure. The composite was opaque and had some light streaks in places where the prepreg resin had little or no flow, as observed during the winding. The surface-resin color was darkened considerably when the composite was postcured at 500°F.

Because of the low quality composite obtained from the initial winding, which was attributed to poor resin flow, an evaluation to determine the resin flow characteristics of all prepregs was

performed by fabrication NOL rings on standard steel mandrels. The mandrels were preheated at 300°F and additional heat was applied, as needed, with a heat gun and quartz strip heater during winding to maintain the resin flow temperature on the mandrel. All windings were made under an 8-pound winding tension and cured at 350°F for 4 hours and postcured at 300°F. The postcured rings were visually examined for translucency or lack of it and related to the resin flow. The results based on these observations indicated that there is a direct relationship of resin flow with prepreg resin content and, to some extent, with volatile content of the prepreg resin. The 20 and 25% resin prepregs showed very poor resin flow. The NOL rings were light colored and opaque, as noted previously with the cylinder fabricated on a plaster mandrel. These compared with a deep translucency of the rings made with the 30 and 35% prepregs. Table X summarizes the results of the resin flow evaluation and observation of the NOL ring fabrication.

It was decided to order additional materials to replace the two marginal prepregs for use in the winding parameter study. For these materials an acceptance requirement was established for prepreg volatile contents at 1.2 to 2.5 weight percent. It was anticipated that the higher volatile content would assist in obtaining the required flow.

The packaging of the two additional spools of prepreg by General Electric was modified in an attempt to prevent material slippage from the ends of the spool, as was experienced previously. Disks were mounted on the ends of a standard 3-inch-diameter cardboard core. The prepregs were wound on a core with a parallel-wound pattern, but the spool was noticeably soft and bulky. It was noted during winding under tension that the material had a tendency to bury itself in the package and cause some abrasion between strands. The condition was aggravated when 12-pound tension was applied. The material had to be

respooled under tension onto another core to achieve a firmer package of roving.

Gemon-L prepregs were characterized for resin content, volatiles, and the weight-per-yard of glass roving. Three samples were taken from each spool. The test data in Table XI shows the volatile content of the two materials met the acceptance requirement of 1.2 to 2.5 weight percent. The volatile contents were 1.65 and 1.75 weight percent, which are equivalent to 6.62 and 7.04% based on the resin.

The resin flow and processing characteristics of the two prepregs were evaluated by fabricating NOL rings on standard steel mandrels. The winding procedure was essentially the same as used previously to check the material resin flow. The mandrels were preheated at 250°F and additional heat was applied, as needed, to maintain the resin flow temperature. The windings were made under an 8-pound winding tension and cured at 350°F for four hours. A desired level of resin flow was obtained with the two materials at 225 to 235°F mandrel temperature. This is compared with a 280°F mandrel temperature used for winding with the previous materials which did not produce sufficient resin flow. With the knowledge that the two materials possessed satisfactory processing characteristics, the fabrication of test cylinders for the winding parameter study was undertaken.

2. Winding Parameter Study

This task involved fabrication of twelve cylinders on plaster mandrels using prepregs with four variations in resin content (nominally 23, 25, 30, and 35 w/o) and winding each material under three levels of winding tension (4, 8, and 12-pound/12-end). The plaster mandrel was preheated at 220 to 230°F and heat was applied to the prepreg and mandrel during winding. The amount of heat applied was

controlled qualitatively based on observation of resin flow and material compaction. Two layers of glass-roving overwrap pressure was applied using the same winding tension and lead as with the prepreg windings.

Some resin exuded out to the surface from the 12-pound-tension composites using the 30 and 35% resin during the 350°F cure. There was no evidence of resin flow from the rest of the composites as the overwrap glass rovings were removed after the primary cure. All composites were similar in appearance; they were dense and translucent with a deep reddish-brown color. The surface resin turned black from oxidation during the 500°F postcure for 18 hours.

Ring tensile test specimens were cut from the cylinders. The cylinder surfaces were initially machined down to 5.950-inch diameter to produce a 0.100-inch wall thickness. The cylinders were sufficiently large to yield 7 rings, 0.250-inch wide. Any large variation in the specimen thickness was corrected by mounting the ring on a special holding fixture and routing the surface in a lathe. Four ring specimens from each cylinder were prepared for testing. One specimen was tested for ring compressive modulus. This ring was subsequently cut radially into segments that were six times the specimen thickness. Two samples were used for composite gravimetric analysis and five samples were treated for horizontal shear strength at ambient temperature. Three rings from each cylinder were tested for tensile strength using a hydraulic-type NOL ring tensile tester. In most cases, the specimen failure occurred catastrophically at the ultimate burst pressure with a clean breakage through the entire composite cross section. A few specimens had a combination of tensile failure and hoop wise fiber delaminations, but the tensile strength was not apparently affected by the different modes of

of composite failure, as indicated by equivalent strength values obtained within a test group. Also, the tensile strengths did not appear to be affected by repeated pressurization which was made necessary in some tests when leakage of the hydraulic fluid past the sealing ring occurred during test.

The processing parameters and results of the evaluation are summarized in Table XII. To facilitate the analysis of the test data, the composite physical and mechanical properties were converted and normalized on the basis of 100% glass and specific composite property-to-composite density ratios properties. These data are also presented in topographic block forms in Figures 2 to 5 to illustrate the responses and any interactions from the variables evaluated. Some of the most significant effects on the mechanical properties are shown in Figures 6 to 8 as a function of composite resin content and in Figures 9 and 10 as a function of winding tension.

The test data shows that resin-content variation had a greater effect on the composite properties than variation in winding tension. Composites with low resin content (high glass-volume fraction) generally yielded high strength values in all the categories of composite properties determined. The data shows a direct relationship of ring modulus with glass-volume fraction over the range of resin content evaluated. The ring modulus varied from 5.9 to 7.6 x 10^6 psi. The moduli, when normalized on the basis of 100% glass, were generally equivalent among the various composites at around 12 x 10^6 psi, which approached very closely to the published value of 12.4 x 10^6 psi modulus of elasticity of virgin S-901 glass filament.

Horizontal shear and tensile strengths increased with glass-volume fraction as noted with ring modulus, but the strengths appeared to peak out at around 24 w/o composite resin. The horizontal

shear strengths ranged from a low of 10.6 ksi (34.5 w/o composite resin) to a high of 14.4 ksi (22.3 w/o composite resin). A similar trend on the effect of resin content was observed when the shear strength values were normalized at 100% glass and also converted to specific strength. The large disparity in shear strengths obtained between the composites made from the 30 and 35% resin prepregs and 23 and 25% resin prepregs (Figures 6 and 9) and the fact that these materials were procured in two separate orders suggests that material lot variations may have some significant effects on the composite properties. This contention, however, has not been verified.

The composite tensile strengths were high ranging from 200.2 to 236.2 ksi. The normalized fiber strengths increased as resin content increased (Figure 7). The apparent high translation of fiber strengths in the high resin content composites may have been due to greater protection from fiber damage afforded by the excess resin. The packaging of the roving used was poor with more fraying noticeable during winding. This condition was also probably responsible for some strength reduction.

Void content of all composites was low at 0.8 to 1.4 volume percent. Filament winding tension appeared to have some effect in lowering the composite voids. This was more apparent when the tension was increased from 4 to 8-pound tension than at 12-pound tension. A trend in lowering of composite resin content was also obtained with an increase in winding tension. There was essentially no change in the composite resin content of 4-pound winding tension from the resin content of the starting prepreg material.

The most significant effect of winding tension on composite properties was obtained on the tensile strength (Figure 9). The responses to winding tension varied with prepregs, but generally the highest tensile strength was obtained at 8-pound winding tension. No significant

effect of winding tension was obtained in the horizontal shear strength. Some increases in the shear strength noted for high-tension composites appear to be a secondary effect resulting from lowering of the composite resin content.

Based on the analysis of the test data, the best combination of filament winding variables resulting in superior overall properties of the composite test specimens made from Gemon-L prepregs appeared to be 24% resin and 8-pound winding tension. The above conditions were selected and planned for use in vessel fabrication.

III. PRESSURE VESSELS

A. DESIGN

The design analysis for the pressure vessel is given in Appendix A. The vessel was 4-inch-diameter by 6-inch-long. The design incorporated the use of two strands of 12-end prepreg roving for the longitudinal winding and one strand of prepreg for the hoop winding. Trial windings were made on a cylindrical plaster mandrel and on a standard 4-inch-diameter vessel, rubber-lined plaster mandrel to determine the prepreg band width and to establish the winding pattern for the vessel fabrication. Gemon-L/12-end, S-glass prepreg roving (material available from previous evaluation study) was used for this task. Sufficient heat was applied on the material during winding to obtain some resin flow. The winding lead and the vessel wrap pattern were selected on the basis of the longitudinal windings which produced essentially no gaps in the adjacent rovings in the cylindrical portion of the vessel. The strand width was 0.052-inch for the material used. This data was utilized in the computer run for the filament wound vessel and metal-liner designs.

For the purpose of the design analysis, a 6-pound winding tension and a 67 volume percent fiber content in composites were used for the preliminary values. Actual values developed from the previous-discussed work were used in the final design. The design criteria are listed in Table XIII. Some of these and other considerations are listed below:

1. Fiber/Matrix - Twelve-end S-glass filaments in continous length and preimpregnated with polyimide resin.
2. Shape - Closed-end cylinder
3. Size - 4-inch-diameter by 6-inch-long

4. Liner - Stainless steel, Type 321 (annealed), of 0.006-inch thickness

5. Winding Pattern - In-plane longitudinal with complementing hoop wraps

6. Winding Tension - 8-pound/12-end strand

7. Fiber Content - 67 volume percent

8. Service Temperatures - 75°F, 600°F, -423°F

9. Burst Pressure - Dictated by minimum wrap thicknesses

Figure 11 presents the 321 stainless steel liner design and Figure 12 the complete pressure-vessel test specimen. Figures 13 and 14 show the vessel design stress-strain curves, and Figure 15 presents the vessel design pressure-strain relationships.

B. METAL LINERS

The stainless steel liners were fabricated according to SCI Drawing 1269288 shown in Figure 11, and SCI Specification 9141-5 given in Appendix B.

Twenty-six 4-inch-diameter by 6-inch-long metal liner assemblies of 0.005 to 0.007-inch nominal thickness were prepared. They are shown in Figure 16. They were subjected to a helium test to verify the leak tightness and weld integrity of the stainless steel liner. Details of the procedure are given in Appendix C. Each liner was pressurized with helium gas to 5 $\pm$ 2 psia in a vacuum chamber, and any leakage was measured with a helium mass spectrometer leak detector (Veeco Leak Detector, Model MS-9AB, manufactured by Vacuum-Electronics Corporation). The test results showed that all liners had leakage rate much less than the allowable leak rate of 1×10^{-5} standard cc/second established for the stainless steel liners for this program. The leakage

rate determined ranged from 1×10^{-9} to 3.3×10^{-6} standard cc/second.

The liner test results are presented in Table XIV, including the physical inspection data for the liner overall length measured between the bosses, diameter, and wall thickness in the cylindrical section, and the weight. The physical dimensions of the liners were very uniform considering the shrinkage problem encountered during welding of the bosses to the stretch-formed, thin-walled liners. The liner lengths varied only 0.076 inch between the two extreme cases. The diameter measured at three places 90^{o} apart in the cylindrical section were essentially equivalent for all liners at 3.937 to 3.942 inch. The liner weight varied between 159.4 and 186.4 grams. These variations were caused by different amounts of material removed during chemical milling operation to bring the wall thickness in the cylindrical section within 4 to 8 mils, and variations in the polar boss weights.

C. MANDREL CASTING PROCEDURE

The ability to use plaster as a means of supporting the thin vessel metal liner was of some concern with regard to filling through the small port opening, the assurance of complete support throughout the liner, and thorough drying of the plaster so that complete mandrel removal would be effected after the fabrication of filament would test specimens. An alternate method considered to support the liner was the use of a hydraulically pressurized mandrel system, which would have been much more complicated.

A preliminary evaluation was conducted to gain some insight as to the problems that would be encountered in various phases of the insitu plaster mandrel fabrication. Plaster was cast in a glass bottle with a small opening. A 3/8-inch-diameter rod was inserted through the

length of the bottle to represent the shaft hole. Another plaster casting was made in a glass jar with a 3/8-inch-diameter hole through a twist cap to observe for any large air occlusion during casting and development of cracks and shrinkage during a drying operation.

The effectiveness of drying with only a small hole through the center of the casting was determined on these glass-lined plasters and a totally-exposed plaster casting as a control. The same batch of plaster mix was used for all the castings. These plasters were placed in an air-circulating oven and exposed to heat at 190, 250, and 350°F. Weight losses were determined at various stages of drying. The data, presented in Table XV, show that equivalent amounts of moisture are driven out after 16 hours at 350°F. At 190°F, the rate of weight loss for the glass-lined plasters was considerably slower than the control sample, which was to be expected.

Development of the plaster casting procedure was continued using a sample 4-inch-diameter by 6-inch-long glass filament wound pressure vessel as a liner. Some difficulties were encountered in filling the vessel cavity completely before the plaster started to set. It was necessary to prepare a more fluid plaster mixture than that used for casting cylindrical plaster mandrels. Also, venting was necessary to allow air to expel freely as the vessel cavity was being filled with plaster. Inserting an ordinary drinking straw through the boss opening provided a satisfactory venting system. A 3/8-inch drive shaft was inserted into the soft plaster and removed as soon as the plaster set. The plaster-supported vessel was dried in an oven at 170 and 350°F. Weight loss from the plaster after 24 hours at 350°F was 32 weight percent which was slightly more than the percent weight loss (27 weight percent) obtained from the earlier evaluation. The data is presented in Table XVI. The results of

this study satisfied us as to the feasibility of this approach.

D. FABRICATION AND TESTING

Prior to final fabrication of the liners, two filament wound pressure vessels were fabricated on two representative trial liners to check out the liner design and assembly techniques. The trial runs also served to establish the vessel fabrication procedures., and to permit making any changes, as required, to the preliminary process specification prepared for fabricating pressure vessel test specimens. The two vessels were tested to ultimate burst pressure. The tests showed that the liners performed satisfactorily and no vessel failures were attributed to liner deficiency.

The liner interior cavity was filled completely with Kerr DMM plaster to provide support to the thin walled liner for filament winding. A winding shaft was inserted through the boss openings and was removed as soon as the plaster set. The plaster was dried for 16 hours at 200°F and 8 hours at 350°F. The small shaft hole through the mandrel was sufficient to remove most of the moisture contained in the plaster. The weight measurements taken showed that over 29% weight loss was obtained from drying of the 31% water added to the plaster.

The liner surfaces were prepared to assure good bonding of the filament wound composites to the metal liner. The liner surfaces were cleaned in an alkaline solution (Prebond 700) and subsequently etched in an acid solution consisting of sulfuric acid, hydrochloric acid, and water. A thin coating of polyimide-resin primer (BR-34 primer, American Cyanamid Company) was applied on the cleaned surfaces and treated at 410°F for 45 minutes. Any blisters of primer coating formed during the thermal treatment were scrapped off before filament winding.

A 25% resin "Gemon-L" prepreg material was used in the winding. The liner mandrel assembly was preheated in an oven at 300°F and additional heat was applied on the material during winding to obtain the desired level of resin flow and compaction. A uniform wrap pattern was obtained from two 12-end strands of longitudinal winding and single-strand hoop winding. Glass-roving overwrap was applied over the wound vessel using the same 8-pound/strand winding tension and wrap pattern as with the prepreg roving. The assembly was cured in an oven for 2 hours at 350°F and post cured for 16 hours at 500°F and 2 hours at 550°F. The cured composite appeared very uniform and dense as the glass overwrap was removed after the 350°F primary cure.

While repositioning the wound vessel for glass overwrap, the first vessel was bumped inadvertently on the pay-off roller at the knuckle area which left a small depression. This depression was still present after the cure. The damage was apparently very significant, as shown by the manner in which the vessel failed cleanly at the damaged area during the hydroburst test, as shown in Figure 17. The vessel burst at 2680 psig, which was lower than the 3100 psi minimum predicted design burst pressure for the test vessel. Figure 18 shows the vessel prior to test.

The second vessel was tested to burst at room temperature. It failed in the hoop windings at the middle of the cylindrical section at 3400 psig pressure. While pressurizing the vessel for burst, a leakage in the fittings occurred at 3040 psi gage pressure and the pressure was dropped down very rapidly to zero pressure. With the pressure drop, the vessel was cycled effectively once to approximately 90% stress level before the final burst, and there was apparently no detrimental effect on the ultimate burst strength. The hoop fiber stress calculated at the burst pressure was 426.5 ksi. This compares favorably with the predicted average fiber stress of 423.9 ksi, and minimum design value of 381.0 ksi.

There was no leakage of the hydraulic test fluid after the specimen failure. The vessel bulged out at the unsupported cylindrical section where the hoop composite had failed and broke away from it, as shown in Figure 19. Examination of the test vessel revealed that the surface resin, which turned black from oxidation during the 550°F postcure, was confined mostly to the outer surface layer. The exposed interior section of the composite showed a deep amber color just like the condition after a 350°F primary cure.

E. VESSEL TEST SPECIMEN FABRICATION AND TESTING

Twenty additional filament wound pressure vessels were fabricated. The vessels were fabricated using essentially the same procedure as the two trial vessels fabricated earlier to check the vessel liners and to establish the fabrication procedures for the vessel test specimens. The only change in the test vessel was an incorporation of two instrumentation tacks on each end of the hoop wrap to provide means of attaching wires to the specimen for measuring longitudinal strain during the burst test. Appendix D describes the fabrication procedure. Fabrication data for these vessels is summarized in Table XVII. Two vessels were tested at ambient temperature with both the hoop and longitudinal strains and vessel internal pressure recorded continuously as the vessels were pressurized to burst. The vessel test specimens failed in the hoop fibers at 3400 and 3300 psig. As noted with the earlier trial vessel, the vessels bulged out at the unsupported cylindrical section. There was no leakage of the hydrualic test fluid after the specimen failure. Calculations of the hoop filament stress at failure were 426.7 and 412.9 ksi for the two vessels. The maximum hoop-fiber strains were 3.1 and 3.5%, respectively. Figures 20 and 21 show the longitudinal and hoop-fiber strains as a function of pressure for single-cycle burst test for the two test specimens.

The increase in the hoop strain was equivalent to 1% strain/minute using a 1000 psig/minute pressurization rate.

The efficiencies of the vessel composite, as expressed in terms of $^{pV}/_{W}$ (burst pressure times vessel volume divided by composite weight) were 0.974 and 0.950 x 10^6 inch. These values are somewhat conservative since the undetermined weight of the polyimide-resin primer applied on the metal liner was considered a part of composite weight.

Test data for the vessels is given in Table XVIII. Figure 22 shows the effect of temperature from -320 to 600°F upon burst strength. Except for the values at 300°F, the data is described by a smooth curve over the entire range of temperature. The burst pressure varied from about 5150 psi at -320 to about 2600 psi at 600°F. The hoop filament stresses were about 25 ksi at -320°F and 322 ksi at 600°F, as shown in Figure 23. At room temperature the hoop filament stress was about 415 ksi. As shown in Figure 24, the vessel eficiency, $^{pv}/_{W}$ varied smoothly between 1.2 and 0.77 inch x 10^6 between -320 and 600°F, respectively.

Testing was also conducted to define the effect of thermal aging at 500°F upon vessel performance. As shown in Figure 25, the burst pressure reduced to 2650 psi after 100 hours and 1650 psi after 500 hours. These are equivalent to about 20 and 50% reduction, respectively.

The effect of thermal cycling is shown in Figure 26. Without prior prestress, vessels were cycled between -320 and 600°F for one hundred cycles. The burst pressure of these vessels averaged about 250 psi which is about 25% less than uncycled bottles. Another vessel cycled similarly after prestress failed at 2400 psi, which is within the scatter band of the previous vessel, and suggests no additional

adverse effect from the prestress.

Although the resin used in Gemon-L is not considered to have very good thermal stability at 500°F, it is possible that the results obtained here were less than the potential capability of this resin. Recent work at SCI (Reference 4), has shown that the thermal stability of Kermid 601 is very sensitive to the type of solvent used in the preparation of its prepregs. For example, with DMF its strength is reduced after only 24 hours and its strength is negligible after 100 hours at 500°F, even though its initial strength is quite good. With N-methyl pyrolidone there is no strength reduction after 100 hours at 500°F. Since Gemon-L prepregs were prepared by a proprietary process, SCI is not aware of the specific solvent used for the prepregs in this program. However, we do know that the solvent used was not N-methyl pyrolidone.

IV. CONCLUSIONS AND RECOMMENDATIONS

The program established the feasibility of applying polyimide resin to filament-wound pressure vessels. With Gemon-L (Keramid 601) static strength equal to epoxy resin was demonstrated at cryogenic and ambient temperatures with an initial strength retention at 600°F of almost 80% of the ambient strength. The latter performance is far superior to that which would be anticipated with epoxies. Strength reduction as a function of thermal ageing at 500°F was more severe than would be anticipated with many polyimides with about 50% of the original room temperature strength being retained after 500 hours. This is still a useful level, but one which should be capable of improvement with polyimides of greater thermal capability than Keramid 601. However, it is important to note that findings that have become available since the work reported here was conducted suggest that the thermal stability of Keramid 601 is very sensitive to the type of solvent used in the preparation of its prepregs. It would be of real interest, considering the fact that Keramid 601 is so successful as a filament-winding resin, to further explore this question.

The results of the studies with P10PA were dissatisfying. This class of polyimides has demonstrated substantially better thermal capability than competing polyimides which polymerize by addition. The problem with P10PA appears to related back to the need to retain significant amounts of residual solvent in its prepregs in order to make it tractable, thereby also negating the potential processing advantages of using it in its imidized state.

It is possible that the in-situ polymerizeable monomeric reactant (PMR) technique developed by Lewis Research Center would contribute towards a solution of this problem. By introducing the lower boiling solvents made possible with this approach, it might be possible to use solvents to assist

processing, but then remove them effectively before imidization temperatures are reached. This would still not remove the problem of eliminating the volatile matter produced during imidization. However, the amount of volumetric change prior to polymerization might be reduced to that point that a practical process could be developed.

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TABLE I

CHARACTERIZATION OF P10PA POLYIMIDE RESIN

Lot Number	Bottle Number	Resin Solids, Weight Percent		
		Sample 1	Sample 2	Average
1 (1)	1	35.2	35.3	35.3
	2	35.3	35.3	35.3
	3	35.6	35.6	35.6
	7	35.6	35.6	35.6
2 (2)	1	36.7	36.8	36.8
	2	36.8	36.7	36.8

(1) TRW's lot number 6698-40, manufacturing date 21 July 1971; solution viscosity N at 20°C = 113 cps

(2) TRW's lot number 6698-40, manufacturing date 15 September 1971; solution viscosity N at 20°C = 228 cps

TABLE II

THERMAL CONDITIONING EVALUATION OF

181 A-100/P10PA PREPREG MATERIAL

Drying Condition		Properties		
Temperature °F	Time Minutes	Volatiles Weight Percent	R. C. Weight Percent	Comments
180	15	43.6	28.0	Surface very wet
	30	40.0	30.0	Surface very wet
	60	36.4	31.6	Top surface dry; wet on the mylar side
200	15	36.1	27.3	Top surface dry; wet on the mylar side
	30	30.1	29.0	Some tacky areas on the mylar side
	60	25.2	28.7	Completely dry; stiff but no resin fracturing. Resin flow on 250°F platen
250	15	23.3	31.0	Completely dry; stiff but resin not brittle. Resin flow on 250°F platen
	30	9.5	29.0	Completely dry; resin brittle; no resin flow on 250°F platen
	60	12.6	29.4	Same as above

TABLE III

CHARACTERIZATION DATA OF P10PA/12-END, S-GLASS

PREPREG ROVINGS FABRICATED BY A DRUM-WINDING PROCESS

Prepreg S/N	Target Resin Content Wt. %	Prepreg Drying Time(1) Minutes	Test Sample Data[3]				Prepreg Manufacturing Data				
			Resin Solids Wt. %	Volatiles, Wt. %		Glass Wt./Yd. g	Glass Roving g	Prepreg g	Resin Pickup Wt. %	Resin Volatiles[4] g	Resin Solids Wt. %
				Resin	Prepreg						
1	25	60	25.7	25.4	8.0	0.3645	48	70.3	31.3	8.0	25.3
2	32	60[2]	33.0	21.8	8.4	0.3642	48	75.8	36.7	8.0	31.2
3	24	45	23.5	27.7	8.3	0.3633	126	178.3	29.3	8.1	23.1
4	20	45	19.2	26.8	6.6	0.3624	126	168.7	25.3	6.8	19.8
5	20	45	16.9	28.2	6.2	0.3655	126	168.1	25.0	7.1	19.4
6	28	45	23.4	31.3	9.6	0.3650	123	187.9	34.5	10.8	26.6
7	28	45	20.4	31.9	8.8	0.3653	124	188.3	34.1	10.9	26.0
8	28	60	18.9	29.5	7.3	0.3632	126	185.0	31.9	9.4	24.8
9	24	60	17.0	29.9	6.7	0.3630	122	171.1	28.7	8.6	22.0
10	24	60	21.8	28.7	7.9	0.3655	125	180.1	30.6	8.8	23.9
11	20	45	14.7	31.5	6.3	0.3631	127	166.4	23.7	7.5	17.5
12	20	45	-	-	-	-	126	163.8	23.1	-	-
13	24	60	22.7	29.8	8.8	0.3653	151	215.1	29.2	8.7	22.5
14	24	60	19.1	30.3	7.7	0.3620	150	218.0	31.2	9.5	24.0
15	28	60	24.0	31.0	9.7	0.3627	150	234.0	35.5	11.0	27.5
16	28	60	18.1	32.5	8.4	0.3621	150	231.4	35.2	11.4	26.8
17	28	60	16.6	31.4	7.1	0.3612	152	229.9	33.9	10.6	26.0
18	20	60	16.6	32.1	7.3	0.3624	151	201.8	25.2	8.1	18.6
19	32	75	21.6	30.0	8.4	0.3625	150	241.4	37.9	11.3	30.0

TABLE III (continued)

CHARACTERIZATION DATA OF P10PA/12-END, S-GLASS
PREPREG ROVINGS FABRICATED BY A DRUM-WINDING PROCESS

(1) Resin-impregnated roving air dried for a minimum of one hour prior to heat drying in an oven at 200°F

(2) Oven-drying temperature of 230°F was evaluated for this batch only

(3) The data is based on an average of three test specimens

(4) Volatile weight was calculated from the prepreg resin weight and the percent volatiles determined from the test samples

TABLE IV

RESULTS OF THE NOL RING FABRICATION[1] PROCESSING STUDY WITH P10PA/12-END, S-GLASS PREPREG ROVING

Specimen Number	Prepreg[2] S/N	Processing Variables: Winding Tension Lb.	Imidization Temperature °F	Imidization Time Hour	Cure Pressure[3] psi	Composite Properties: Ring Modulus 10^6 psi	Horizontal Shear ksi	Resin Content Wt. %
1-19	7	8	250	8	50	9.40	6.39	12.5
2-11	7	8	250	16	50	9.56	5.92	12.7
3-74	7	8	250	16	500	9.39	4.47	11.9
4-7	7	8	250	24	50*	8.47	5.08	13.0
5-94	7	8	250	24	1.000	8.68	4.98	15.6
6-77	7	8	350	4	50	9.79	4.76	14.4
7-89	6	8	350	10	50	8.96	6.38	12.8
8-73	6	8	350	10	500	9.53	6.50	13.0
9-37	6	8	350	16	50	5.17[4]	4.75	15.1
10-12	6	4	250	24	50*	7.45	4.83	18.7
11-16	6	4	250	24	50	8.03	4.86	18.5
12-8	6	12	250	24	50*	8.78	5.70	14.0
13-86	6	12	250	24	50	8.82	4.49	17.0
14-29	7	8	250	24	-	10.00	5.87	12.3

(1) NOL rings were cured at 400°F for 2 hours and 4 hours at 550°F after the imidization treatment and overwrap - pressure application. Steel mandrels were preheated at 250°F at the start of winding.

(2) The volatiles and resin - solids content prepregs are:

	Volatiles Weight %	Resin Solids Weight %
S/N 6	31.3	26.6
S/N 7	31.9	26.0

(3) Cure pressure was applied by means of a glass roving overwrap using a 20-end glass roving. The pressure was applied following the imidization treatment, except for the three rings indicated by (*) in which the pressure was applied prior to the imidization treatment.

(4) Some delaminations were visible from the side of the ring.

TABLE V

CHARACTERIZATION DATA OF IMIDIZED, FILAMENT WOUND
P10PA/12-END, S-GLASS PREPREG ROVING

Specimen Number	Prepreg Property[1]		Imidization		Imidized Prepreg Property					
					Resin Content, Weight %			Volatiles, Weight %		
	R.C. Weight %	Volatiles Weight %	Temperature °F	Time Hour	Entire Sample	Outer Half	Inner Half	Entire Sample	Outer Half	Inner Half
15-19	24.0	26.6	250	4	22.2	24.0	18,5	17.6	16.0	21.0
					21.9			17.7		
16-13	23.8	26.9	250	8	22.4	24.6	20.2	14.7	13.0	17.3
					22.6			14.8		
17-86	23.4	27.5	250	12	21.0			11.5		
18-8	19.5	28.0	325	4	21.2	24.3	18.2	15.1	13.5	18.4

(1) Prepreg S/N 3 (target R. C. of 24%) was used for this study; one prepreg sample was taken after winding the NOL ring sample

TABLE VI

FILAMENT WINDING PROCESSING PARAMETERS FOR P10PA/12-END, S-GLASS PREPREG ROVING

Specimen Number	Prepreg[1] S/N	Winding Tension Lb.	Imidization			Cure Pressure	
			Temperature °F	Time Hour	Vacuum Bag	Overwrap[2] psi	Vacuum Bag
20-7	3	8	250	4	No	135*	No
21-11	3	8	250	4	No	135	No
22-86	14	8	250	24	No	135	No
23-94	14	8	250	4	Yest	No	Yes
24-73	14	8	250	24	Yes	No	Yes
25-13	14	8	250	4	Yes	135	Yes
26-1	14	8	250	24	Yes	135	Yes
27-16	14	8	250	24	No	500	No
28-74	14	8	250	8	Yes	No	Yes
30-12	9	8	250	4	Yes	No[3]	Yes
31-77	9	8	250	8	Yes	No[3]	Yes
32-7	9	8	250	24	Yes	No[3]	Yes

(1) Properties of prepreg roving used in the specimen preparation:

S/N	Volatile Weight %	Resin Solids Weight %
3	27.7	23.1
9	29.9	22.0
14	30.3	24.0

(2) The overwrap pressure was applied following the imidization treatment, except for Specimen 20-7*, in which the pressure was applied prior to the imidization treatment.

(3) Mandrel groove was filled with glass-roving overwind to transmit a normal load exerted by the vacuum bag pressure during imidization and cure.

TABLE VII

RESULTS OF THE NOL RING FABRICATION[1] PROCESSING STUDY WITH PI0PA/12-END, S-GLASS PREPREG ROVING

	Composite Properties						
Specimen Number	Specific Gravity	R. C.[2] Weight %	Voids Volume %	Glass Volume %	Modulus, 10^6 psi Ring	Modulus, 10^6 psi Glass	Horizontal Shear Strength ksi
20-7	2.02	18.4	5.6	70.2	8.87	12.63	5.41
21-11	2.03	17.3	6.0	71.7	7.87	10.97	6.21
22-86	(3)						
23-94	2.04	17.0	5.8	72.1	8.01	11.11	7.17
24-73	2.03	19.1	4.8	69.2	8.02	11.58	6.52
25-13	2.02	17.2	4.7	71.8	8.03	11.18	7.42
26-1	2.02	20.7	3.9	66.9	7.86	11.74	4.52
27-16	2.04	20.8	2.9	66.8	8.09	12.11	5.88
28-74	2.02	20.2	4.4	67.6	7.90	11.68	5.22
30-12	2.06	14.7	6.3	75.5	7.95	10.53	5.30
31-77	2.06	15.1	6.1	74.8	8.16	10.90	6.63
32-7	(3)						

(1) See Table V for the processing parameters used in the NOL ring specimen fabrication. The NOL rings were cured at 550°F for 4 hours following the imidization treatment.

(2) Resin content

(3) The specimens had very poor composite integrity; no further tests were performed.

TABLE VIII

TEST RESULTS ON P10PA/12-END, S-GLASS FILAMENT-WOUND COMPOSITES

Prepreg Material					Composite Properties[1]			
Specimen Number	S/N	Resin Weight %	Volatiles Weight %	Winding Tension Lb.	Resin Content Weight %	Ring Modulus 10^6 psi	Shear Strength ksi	Tensile Strength ksi
C 14	20	26.1	31.4	4	21.7			
C 13	19	30.0	29.8	8	22.0			
C 15	21	30.4	32.1	12	20.5			
C 17	16	26.8	32.5	4	23.3			
C 16	15	23.8	31.0	8	24.6			
C 18	17	26.1	31.4	12	21.5	7.10		189.3
C 20	9	22.0	20.0	4	21.1			
C 19	13	22.5	29.8	8	21.6			
C 21	23	20.5	30.0	12	20.4	7.65	2.30	198.0
C 23	25	33.2	28.4	4	32.0			
C 22	24	33.9	26.0	8	26.6			
C 24	26	31.6	28.1	12	24.3			

(1) Only the composites that had sufficient integrity were tested for mechanical properties, as shown

TABLE IX
CHARACTERIZATION DATA OF GEMON-L/12-END,
S-GLASS PREPREG ROVING

Roll No.	PO Requirements R. C. Wt. %	PO Requirements Vol Wt. %	GE Data (3) R. C. Wt. %	GE Data (3) Vol. Wt. %	SCI Data R. C. Wt. %	SCI Data Volatiles, Wt. % Prepreg	SCI Data Volatiles, Wt. % Resin	SCI Data Wt/Yd g
1	20 ± 2	< 2	19.9	1.5	20.1	0.46	2.25	0.3610
					20.5	0.44	0.3620	
					20.5	0.46	2.20	0.3620
					20.0 (1)	0.40	1.89	0.3603
					20.6 (1)	0.42	1.99	0.3600
					20.6 (1)	0.33	1.58	0.3604
			Average		20.4	0.42	2.00	0.3610
2	25 ± 2	< 2	25.1	1.5	27.2	0.24	0.88	0.3640
					27.6	0.29	1.04	0.3630
					27.1	0.29	1.03	0.3630
					23.7 (1)	0.49	2.04	0.3620
					23.6 (1)	0.47	1.97	0.3626
					23.6 (1)	0.54	2.27	0.3625
			Average		25.5	0.38	1.54	0.3628
3	30 ± 2	< 2	29.3	1.5	26.9	0.09	0.34	0.3633
					26.5	0.01	0.23	0.3635
					29.1	0.67	2.26	0.3636
			Average		27.5	0.26	0.94	0.3635
4	30 ± 2	< 2	29.3	1.5	31.0	0.79	2.49	0.3610
					29.5	0.68	2.26	0.3615
					30.0	0.64	2.09	0.3607
			Average		30.2	0.70	2.28	0.3611
5	35 ± 2	< 2	33.6	1.8	34.6	1.26	3.55	0.3626
					34.4	1.22	3.45	0.3625
					34.7	1.45	4.06	0.3624
			Average		34.6	1.31	3.69	0.3625
6	35 ± 2	< 2	33.6	1.8	34.0	0.25	0.75	0.3620
					33.6	0.75	2.22	0.3628
					35.0	0.41	1.17	0.3624
			Average		34.2	0.47	1.38	0.3624
A (2)	35 ± 4	< 2			26.5	1.17	4.27	0.3641
					26.3	1.28	4.69	0.3647
					26.3	1.12	4.12	0.3646
			Average		26.4	1.19	4.36	0.3645
B (2)	25 ± 4	< 2			26.6	1.15	4.20	0.3625
					27.5	1.16	4.10	0.3586
					27.2	1.18	4.37	0.3615
			Average		27.4	1.16	4.22	0.3608

(1) Samples from the end of the roll after rewind
(2) Materials procured in October 1970
(3) Volatiles based on prepreg weight

TABLE X

GEMON-L PREPREG RESIN FLOW EVALUATION BY FABRICATION [1] OF NOL RINGS

Specimen Number	Prepreg Material: Spool Number	R. C. WT. %	Vol. Wt. %	Winding Tension lb.	Mandrel Temp. °F	Resin Flow Observation
1 - 39	1	20.5	0.4	8	280	Very little flow; light straw color
2 - 8	2	25.5	0.4	8	285	Some local flow; light color; streaks
3 - 16	3	27.5	0.2	8	280	Good flow; some surface resin beads
4 - 79	4	30.2	0.7	8	290	Good flow; some excess surface resin
5 - 74	5	33.5	1.3	8	270	Excess flow out to surface
6 - 81	6	33.5	0.5	8	290	Excess flow out to surface
A - 94	A	26.3	1.2	8	290	Good flow; excess flow out

(1) NOL rings were cured at 350° for 4 hours and postcured at 500°F for 18 hours; observations of the cured rings were as follows:

Specimen Number	Observation of Cured NOL Rings
1 - 39	Light straw color overall; no resin flow; poor composite
2 - 8	Some local resin flow; surface appeared dry; some light streaks in composite
3 - 16	Resin barely flowed out to the surface; some translucency but not as deep and uniform as the specimens below
4 - 79	Thin surface resin layer; deep translucency and uniform overall - very good composite appearance
5 - 74	Overall appearance very much like 4 - 79 above
6 - 81	Excessive surface resin; overall appearance very much like 4 - 79 and 5 - 74
A - 94	Good resin flow out to surface and formation of thin resin layer; deep translucent and uniform composite

TABLE XI

CHARACTERIZATION DATA OF GEMON-L/12-END,
S-GLASS PREPREG ROVINGS

Roll Number	PO Requirement R. C. Wt. %	PO Requirement Vol.[1] Wt. %	GE Data R. C. Wt. %	GE Data Vol.[2] Wt. %	SCI Data R. C. Wt. %	SCI Data Volatiles, Wt. % Prepreg	SCI Data Volatiles, Wt. % Resin	SCI Data Wt/Yd[3] g
1a	20 ± 2	1.1-2.5	22.4	2.2	24.8	1.97	7.37	0.3610
					22.5	1.47	6.22	0.3635
					23.2	1.52	6.26	0.3628
			Average		23.5	1.65	6.62	0.3624
2a	25 ± 2	1.1-2.5	25.0	2.5	25.4	1.82	6.82	0.3612
					25.7	1.74	6.43	0.3610
					25.5	1.70	6.37	0.3610
			Average		25.5	1.75	6.54	0.3611

(1) Volatiles based on prepreg weight

(2) Resin content

(3) Glass roving after ignition loss

TABLE XII

SUMMARY OF TESTS RESULTS ON GEMON L/12-END, S-GLASS FILAMENT-WOUND COMPOSITES

Prepreg Material					Composite Gravimetric Data					Composite Properties			Fiber Properties(1)			Specific Composite Properties(2)		
Specimen No.	Spool No.	(3) R.C. Wt%	(4) Vol Wt%	Winding Tension, lb	Specific Gravity	Density lb/in^3	(3) R.C. Wt%	Voids Vol%	Glass Content Vol %	Shear Strength ksi	Tensile Strength ksi	Ring Modulus 10^6 psi	Shear Strength ksi	Tensile Strength ksi	Modulus 10^6 psi	Shear Strength 10^3 in.	Tensile Strength 10^6 in.	Ring Modulus 10^6 in.
C2	5	34.6	1.31	4	1.863	0.06731	34.5	1.2	49.6	10.6	204.1	5.93	21.3	411.5	11.96	157.1	3.03	88.1
C1				8	1.907	0.06890	31.5	1.0	53.0	11.7	206.2	6.40	22.1	389.1	12.07	169.8	2.99	92.9
C3				12	1.908	0.06894	31.5	0.9	53.0	11.9	200.2	6.24	22.4	377.7	11.77	171.9	2.90	90.5
C5	4	30.2	0.70	4	1.913	0.06912	30.4	1.4	54.3	10.8	218.9	6.48	19.8	403.1	11.95	155.5	3.17	93.8
C4				8	1.923	0.06948	29.9	1.3	54.9	11.6	226.0	6.56	21.1	411.7	11.95	166.6	3.25	94.4
C6				12	1.947	0.07035	28.4	1.1	56.6	11.9	209.7	6.83	21.0	370.5	12.06	168.8	2.98	97.1
C8	2a	25.5	1.75	4	2.005	0.07244	24.4	1.2	61.5	14.3	213.4	7.17	23.2	347.0	11.65	196.7	2.95	99.0
C7				8	2.007	0.07251	24.7	0.8	61.2	14.3	236.2	7.34	23.3	385.9	12.00	197.0	3.26	101.2
C9				12	2.024	0.07313	23.6	0.9	62.6	14.3	217.9	7.58	22.8	348.1	12.11	195.5	2.98	103.7
C11	1a	23.5	1.65	4	2.018	0.07291	23.4	1.3	63.0	13.5	229.0	7.43	21.5	363.5	11.81	185.4	3.14	101.9
C10				8	2.032	0.07342	22.9	1.0	63.6	14.4	226.0	7.42	22.6	355.3	11.67	195.8	3.08	101.1
C12				12	2.035	0.07352	22.7	1.0	63.8	13.9	211.3	7.47	21.7	331.2	11.72	188.7	2.87	101.6

(1) Fiber properties determined on the basis of 100% glass, eg. fiber strength = $\frac{\text{composite strength}}{\text{glass vol. fraction}}$

(2) Specific composite properties determined from the composite property divided by the composite density.

(3) Resin content

(4) Volatile content based on prepreg weight

TABLE XIII

DESIGN CRITERIA

4-INCH-DIAMETER BY 6-INCH-LONG 321 STAINLESS STEEL LINED GLASS FILAMENT WOUND PRESSURE VESSELS

Geometry and Loading

Diameter, inch	3.942
Length, inch	5.640
Polar Boss Diameter, inch	0.840
Metal Liner Thickness, inch	0.006
Design Burst Pressure at 75°F, psig*	3,100
Winding Tension, pounds/12-end	8.0

Material Properties

	Type 321 Stainless Steel Annealed	Glass Filament Wound Composite
Density, pound/inch^3	0.289	0.072
Coefficient of thermal expansion, inch/inch - °F at +75 to +600°F	9.50×10^{-6}	2.010×10^{-6}
Tensile-yield strength, psi	38,000	-
Derivative of yield strength with respect to temperature, psi/°F	-13.0	-
Elastic modulus, psi	28.0×10^6	$12.4 \times 10^{6***}$
Derivative of elastic modulus with respect to temperature, psi/°F	-8,030	-2,410
Plastic modulus, psi	384,000	-
Derivative of plastic modulus with respect to temperature, psi/°F	-0.1	-
Poisson's ratio	0.295	-
Derivative of Poisson's ratio with respect to temperature, 1/°F	0.0	-
Volume fraction of filament in composite	-	0.67**
Hoop filament, design allowable stress at 75°F, psi	-	390,000

* Determined from analysis of other design factors

** Preliminary value

*** Filament modulus

TABLE XIV

METAL LINER INSPECTION DATA

Liner S/N	Length[1] inch	Diameter[2] inch	Thickness[3] 10^{-3} inch	Weight gram	Helium Leak Rate[4] Std cc x 10^{x}/sec.
2	6.280	3.938	7.1	186.4	1.0 (x = -8)
3A	6.324	3.938	7.0	177.8	3.3 (x = -6)
5A	6.280	3.938	6.8	173.7	3.0 (x = -6)
6A	6.298	3.938	7.0	166.3	1.0 (x = -6)
7A	6.278	3.939	5.7	172.0	1.3 (x = -6)
8A	6.305	3.938	6.9	180.6	1.2 (x = -7)
9A	6.288	3.939	5.3	165.7	1.3 (x = -7)
10A	6.304	3.939	6.1	167.0	2.4 (x = -7)
11A	6.290	3.940	6.9	171.9	4.0 (x = -7)
12A	6.257	3.940	6.9	175.9	4.0 (x = -8)
13A	6.288	3.939	6.1	168.0	2.4 (x = -8)
14A	6.293	3.937	6.8	175.7	2.0 (x = -8)
15A	6.283	3.937	5.8	160.6	1.2 (x = -7)
16A	6.298	3.937	5.8	166.1	5.6 (x = -8)
17A	6.291	3.937	6.6	168.7	5.6 (x = -7)
18A	6.304	3.940	7.0	174.8	4.0 (x = -7)
19A	6.294	3.940	6.0	180.0	4.0 (x = -7)
20A	6.254	3.938	6.4	174.6	8.0 (x = -8)
21A	6.273	3.947	6.4	165.1	3.0 (x = -7)
22A	6.265	3.939	6.8	170.0	3.0 (x = -7)
23A	6.278	3.942	6.3	167.3	3.0 (x = -7)
24A	6.330	3.940	5.7	180.0	3.0 (x = -7)
25A	6.270	3.940	5.5	164.4	2.0 (x = -7)
27A	6.264	3.937	6.4	166.0	8.5 (x = -8)
28A	6.254	3.939	5.7	159.4	3.3 (x = -7)
29A	6.285	3.940	5.8	167.4	1.2 (x = -7)

(1) Overall length was measured between the bosses.
(2) Diameter was measured at 6 places in the middle and at the tangency points 90^{o} apart.
(3) Thicknesses were measured at 12 places in the middle and at the tangency points 90^{o} apart in the cylindrical sections of the liners.
(4) Maximum acceptable leak rate was established at 1×10^{-5} std cc/sec.

TABLE XV

PLASTER MANDREL DRYING EVALUATION

		Plaster In Soda Bottle		Plaster In Glass Jr		Plaster All Exposed	
		Sample Weight	Percent Weight Loss	Sample Weight	Percent Weight Loss	Sample Weight	Percent Weight Loss
Tare Weight, g		431.7		225.2			
Tare plus Plaster, g		1,080.6		896.6			
Wet Plaster, g		648.9(1)		671.4(1)		749.1	
Drying:							
Room Temperature:	2 hours	1,080.5	-	896.3	-	738.0	1.5
190°F:	16 hours	1,070.4	1.6	886.0	1.6	570.8	22.6
250°F:	8 hours	932.8	22.7	798.2	14.6	547.6	25.7
350°F:	16 hours	906.4	26.9	712.8	27.5	539.9	26.8

(1) 3/8-inch-diameter hole in the middle of the plaster running parallel with the axis of the cylindrical container

TABLE XVI

PLASTER MANDREL DRYING EVALUATION

	Sample Weight g	Percent Weight Loss
Vessel	139.4	
Vessel plus Wet Plaster	1,615.0	
Wet Plaster(1)	1,475.6	
Drying:		
170°F: 16 hours	1,549.0	4.5
350°F: 8 hours	1,144.0	31.9
24 hours	1,129.0	32.9

(1) 3/8-inch-diameter hole in the middle of the plaster running through both ends of the vessel

TABLE XVIII

PRESSURE VESSEL FABRICATION DATA

Vessel S/N	Metal Liner S/N	Weight, grams: Metal Liner	Primer Coating	Instrumentation Tacks	Roving Deposited: Longo	Roving Deposited: Hoop	Roving Deposited: Total	Cured Vessel	Composite Mat'l[2]	Cylinder Outside Diameter, inch: Prior to Overwrap	Cylinder Outside Diameter, inch: Completed Vessel	Vessel Overall Length, inch: Prior to Overwrap	Vessel Overall Length, inch: Completed Vessel	Vessel Volume inch^3	Burst Pressure psig	Test Temp °F	Pv/W in x 10^6	Hoo Stra ϵ_θ
1	28A	159.6	(1)	2.1	55.3	32.5	87.8	252.0	90.3[3]	3.939	4.012	6.254	6.234	56.95	3400	R T	0.97	.031
2	5A	173.7	(1)	2.1	57.0	33.1	90.1	266.2	90.4[3]	3.938	4.017	6.278	6.257	57.30	3300	R T	0.95	.035
3	6A	166.3	(1)	2.1	55.6	33.4	89.0	257.3	88.9[3]	3.938	4.013	6.298	6.276	56.86	3800	-320	1.10	.032
4	9A	165.7	(1)	2.0	55.2	33.1	88.3	257.5	89.8[3]	3.939	4.014	6.288	6.268	57.35	4150	-320	1.20	.033
5	10A	167.0	(1)	2.1	57.1	32.0	89.1	259.6	90.5[3]	3.939	4.014	6.304	6.280	57.35	2800	500	0.81	.03(1
6	14A	175.7	(1)	2.0	55.3	32.6	87.9	267.0	89.3[3]	3.937	4.017	6.293	6.268	57.23	2850	500	0.83	.023
7	3A	177.8	1.7	1.4	57.4	33.5	90.9	269.0	88.1	3.938	4.013	6.324	6.300	57.35	Leaked	Stressed[4] & Cycled	-	-
8	7A	172.0	1.7	1.6	58.2	32.6	90.8	264.4	89.1	3.939	4.014	6.278	6.260	57.41	2720	600	0.80	.021
9	8A	180.6	2.0	1.5	56.5	33.1	89.6	272.3	88.2	3.938	4.013	6.305	6.277	57.66	2725	Cycled/[5] R T	0.81	.020
10	11A	171.9	2.0	1.4	56.6	32.3	88.9	262.7	87.4	3.940	4.017	6.290	6.266	57.66	2830	300	0.85	.025
11	12A	175.9	1.4	1.5	56.5	32.2	88.7	266.3	87.5	3.940	4.015	6.257	6.238	57.35	2650	100[6] Aged/RT	0.79	
12	13A	168.0	1.0	1.5	55.4	32.8	88.2	258.4	87.9	3.939	4.010	6.288	6.265	57.35	3400	R T	1.01	.027
13	15A	160.6	1.7	1.6	56.0	31.9	87.9	250.9	87.0	3.937	4.012	6.283	6.263	57.47	4250	-320	1.27	.038
14	16A	166.1	2.0	1.5	55.2	32.5	87.7	257.2	87.6	3.937	4.018	6.298	6.273	57.60	3100	-300	0.93	-
15	17A	168.7	2.0	1.5	56.3	32.4	88.7	260.5	88.3	3.937	4.014	6.2	6.262	57.35	1650	500[7] Aged/RT	0.49	
16	18A	174.8	1.7	1.5	56.3	32.8	89.1	266.2	88.2	3.940	4.016	6.304	6.275	57.66	-[9]	-	-	-
17	19A	180.0	2.4	1.4	55.2	32.4	87.6	268.1	84.3	3.940	4.016	6.294	6.269	57.60	2350	500[8]	0.73	.027
18	20A	174.6	2.0	1.4	54.9	32.6	87.5	264.8	86.8	3.939	4.011	6.254	6.230	57.29	2740	500[8]	0.82	.029
19	21A	165.1	1.7	1.4	56.2	32.6	88.8	255.9	87.7	3.937	4.008	6.273	6.249	57.35	2200	-300	0.65	-
20	22A	170.0	2.0	1.4	57.6	32.8	90.4	263.7	90.3	3.939	4.017	6.265	6.249	57.42	4150	-320	1.20	.036
21	23A	167.3	2.0	1.4	57.0	32.1	89.4	259.1	88.4	3.942	4.011	6.278	6.256	57.42	2350	Cycled/[5] R T	0.69	.020
22	24A	180.0	1.7	1.4	55.3	32.7	88.0	269.7	86.6	3.940	4.017	6.330	6.304	57.90	2450	600	0.74	.023
23	25A	164.4	2.5	1.4	54.7	31.7	86.4	253.7	86.2	3.940	4.017	6.270	6.248	57.35	2360	Stressed[6] Cycle/RT	0.71	.027
24	27A	166.0	1.7	1.4	55.7	32.6	88.3	256.7	87.6	3.937	4.013	6.264	6.248	57.60	2600	R T	0.78	.023
25	29A	167.4	2.5	1.4	54.8	32.0	86.8	257.3	86.0	3.940	4.014	6.285	6.262	57.35	-[9]	-	-	-
26	2	186.4	2.0	1.4	55.5	31.8	87.3	275.6	85.8	3.938	4.013	6.280	6.256	57.84	2480	300	0.76	.023

(1) Primer weight was not recorded

(2) Composite Weight = cured vessel weight minus liner, primer, and instrumentation tack weights

(3) Composite includes the weight of primer whose data was not recorded

(4) Pre-stressed to 2000 psig (60% ultimate) then thermal cycled -320° to 600°F for 100 cycles; tested at RT

(5) Thermal cycled -320° to 600°F for 100 cycles; tested at RT

(6) Thermal ageing for 100 hours at 500°F; te

(7) Thermal ageing for 500 hours at 500°F; te

(8) Pre-stressed at 2000 psig (60% ultimate) tested at 500°F

(9) Liner separated and blistered when subjec

(10) Erroneous strain data obtained

DATA

Vessel Overall Length, inch								ksi				
Prior to Overwrap	Completed Vessel	Vessel Volume inch^3	Burst Pressure psig	Test Temp $^\circ$F	Pv/W in x 10^6	Hoop Strain ϵ_θ	Longo Strain ϵ_ϕ	Liner Hoop Stress σ_θ	Liner Longo Stress σ_ϕ	Fiber Hoop Stress $\sigma_{f,h}$	Fiber Longo Stress $\sigma_{f,l}$	Vessel S/N
6.254	6.234	56.95	3400	R T	0.97	.0314	.0262	60.0	58.7	426.7	316.1	1
6.278	6.257	57.30	3300	R T	0.95	.0353	.0232	61.3	58.2	412.9	306.0	2
6.298	6.276	56.86	3800	-320	1.10	.0323	.0163	57.98	53.88	480.5	360.9	3
6.288	6.268	57.35	4150	-320	1.20	.0336	-	54.48	43.31	528.2	404.1	4
6.304	6.280	57.35	2800	500	0.81	.03(Est)	.02		55.19	349.6	255.9	5
6.293	6.268	57.23	2850	500	0.83	.0232	.0081	51.22	47.36	357.3	266.2	6
6.324	6.300	57.35	Leaked	Stressed & Cycled(4)	-	-	-	-	-	-	-	7
6.278	6.260	57.41	2720	600	0.80	.0215	.0065	49.43	45.08	340.8	254.1	8
6.305	6.277	57.66	2725	Cycled/(5) R T	0.81	.0201	.0166	51.81	50.92	340.5	250.9	9
6.290	6.266	57.66	2830	300	0.85	.0258	.0210	55.86	54.63	352.8	259.4	10
6.257	6.238	57.35	2650	100(6) Aged/RT	0.79							11
6.288	6.265	57.35	3400	R T	1.01	.0275	.0263	58.09	57.78	427.4	316.7	12
6.283	6.263	57.47	4250	-320	1.27	.0383	.0350	65.84	65.00	537.0	400.6	13
6.298	6.273	57.60	3100	-300	0.93	-(10)	-(10)	-	-	388.8	288.7	14
6.2	6.262	57.35	1650	500(7) Aged/RT	0.49							15
6.304	6.275	57.66	-(9)	-	-	-	-	-	-	-	-	16
6.294	6.269	57.60	2350	500(8)	0.73	.0272	.0205	56.45	54.73	289.0	209.4	17
6.254	6.230	57.29	2740	500(8)	0.82	.0298	.0199	57.62	55.09	340.2	249.8	18
6.273	6.249	57.35	2200	-300	0.65	-(10)	-(10)	-	-	269.5	195.0	19
6.265	6.249	57.42	4150	-320	1.20	.0361	.0278	63.23	58.31	524.8	394.5	20
6.278	6.256	57.42	2350	Cycled/(5) R T	0.69	.0201	.0189	52.40	52.10	290.6	211.1	21
6.330	6.304	57.90	2450	600	0.74	.0234	.0186	54.10	52.79	303.2	221.0	22
6.270	6.248	57.35	2360	Stressed(6) Cycle/RT	0.71	.0275	.0179	55.94	53.48	290.5	211.2	23
6.264	6.248	57.60	2600	R T	0.78	.0230	.0144	52.74	50.53	323.6	238.1	24
6.285	6.262	57.35	-(9)	-	-	-	-	-	-	-	-	25
6.280	6.256	57.84	2480	300	0.76	.0230	.0220	54.68	54.42	306.9	223.1	26

(6) Thermal ageing for 100 hours at 500°F; tested at RT

(7) Thermal ageing for 500 hours at 500°F; tested at RT

(8) Pre-stressed at 2000 psig (60% ultimate) then thermal cycled -320° to 600°F for 6 cycles; tested at 500°F

(9) Liner separated and blistered when subjected to thermal cycling; not burst test performed

(10) Erroneous strain data obtained

FIGURE 1: Spools of Gemon L Prepreg Roving

Voids, vol %

Prepreg Resin Content, wt % \ Winding Tension, lb	4	8	12
35	1.2	1.0	0.9
30	1.4	1.3	1.1
25	1.2	0.8	0.9
23	1.3	1.0	1.0

Specific Gravity

Prepreg Resin Content, wt % \ Winding Tension, lb	4	8	12
35	1.86	1.91	1.91
30	1.91	1.92	1.95
25	2.01	2.01	2.02
23	2.02	2.03	2.04

Resin Content, wt %

Prepreg Resin Content wt % \ Winding Tension, lb	4	8	12
35	34.5	31.5	31.5
30	30.4	29.9	28.4
25	24.4	24.7	23.6
23	23.4	22.9	22.7

Glass Content, vol %

Prepreg Resin Content wt % \ Winding Tension, lb	4	8	12
35	49.6	53.0	53.0
30	54.3	54.9	56.6
25	61.5	61.2	62.6
23	63.0	63.0	63.8

FIGURE 2: Composite Physical Properties of Gemon L/12-End, S-Glass NOL Rings

Horizontal Shear Strength, Ksi

Prepreg Resin Content, wt %	4	8	12
35	10.6	11.7	11.9
30	10.8	11.6	11.9
25	14.3	14.3	14.3
23	13.5	14.4	13.9

Winding Tension, lb

Tensile Strength, Ksi

Prepreg Resin Content, wt %	4	8	12
35	204.1	206.1	200.2
30	218.9	226.0	209.7
25	213.4	236.2	217.9
23	229.0	226.0	211.3

Winding Tension, lb

Ring Modulus, 10^6 psi

Prepreg Resin Content wt %	4	8	12
35	5.93	6.40	6.24
30	6.48	6.56	6.86
25	7.17	7.34	7.58
23	7.43	7.42	7.47

Winding Tension, lb

FIGURE 3: Composite Mechanical Properties of Gemon L/12-End, S-Glass NOL Rings

Horizontal Shear Strength, Ksi

Prepreg Resin Content, wt %	4	8	12
35	21.3	22.1	22.4
30	19.8	21.1	21.0
25	23.2	23.3	22.8
23	21.5	22.6	21.7

Tensile Strength, Ksi

Prepreg Resin Content, wt %	4	8	12
35	411.5	389.1	377.7
30	403.1	411.7	370.5
25	347.0	385.9	348.1
23	363.5	355.3	331.2

Winding Tension, lb

Ring Modulus, 10^6 psi

Prepreg Resin Content, wt %	4	8	12
35	11.96	12.07	11.77
30	11.95	11.95	12.06
25	11.65	12.00	12.11
23	11.81	11.67	11.72

Winding Tension, lb

FIGURE 4: Normalized Fiber Properties of Gemon L/12-End, S-Glass NOL Rings

Horizontal Shear Strength, 10^3 in.

Prepreg Resin Content, wt %	4	8	12
35	157.1	169.8	171.9
30	155.5	166.6	168.8
25	196.7	197.0	195.5
23	185.4	195.8	188.7

Winding Tension, lb

Tensile Strength, 10^6 in.

Prepreg Resin Content, wt %	4	8	12
35	3.03	2.99	2.90
30	3.17	3.25	2.98
25	2.95	3.26	2.98
23	3.14	3.08	2.87

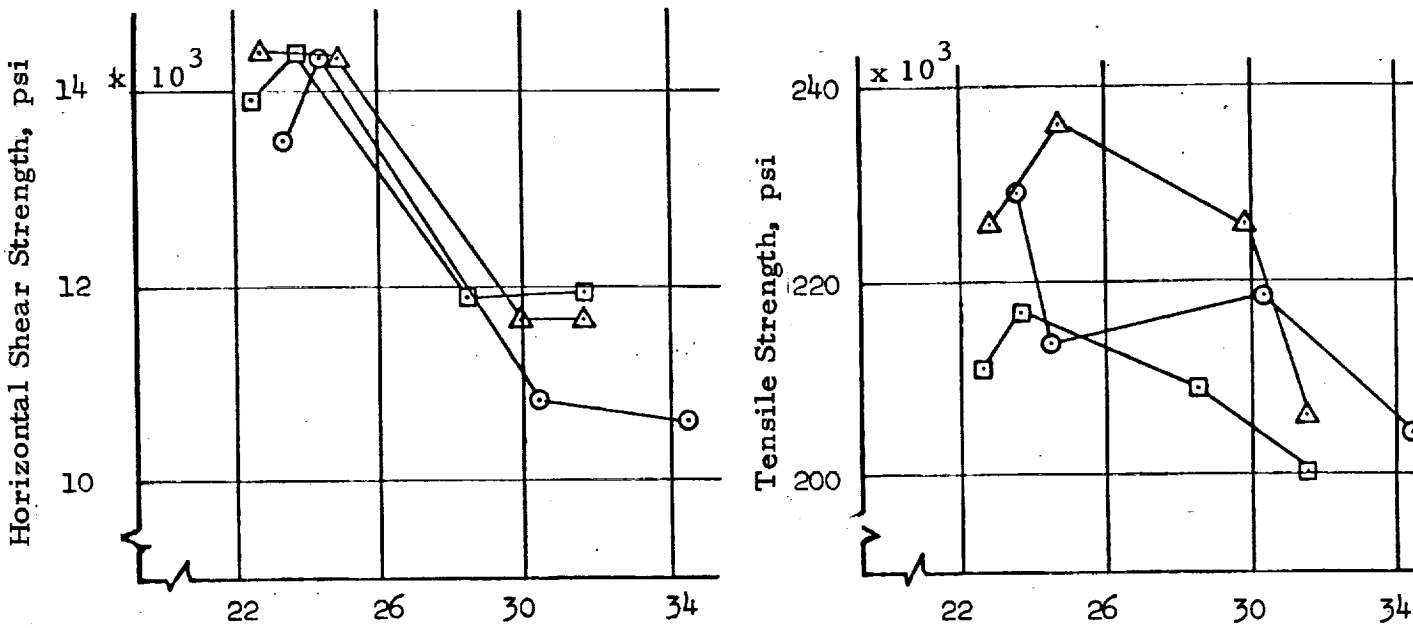
Winding Tension, lb

Ring Modulus, 10^6 in.

Prepreg Resin Content, wt %	4	8	12
35	88.1	92.9	90.5
30	93.8	94.4	97.1
25	99.0	101.2	103.7
23	101.9	101.1	101.6

Winding Tension, lb

FIGURE 5: Specific Properties of Gemon L/12-End, S-Glass NOL Rings



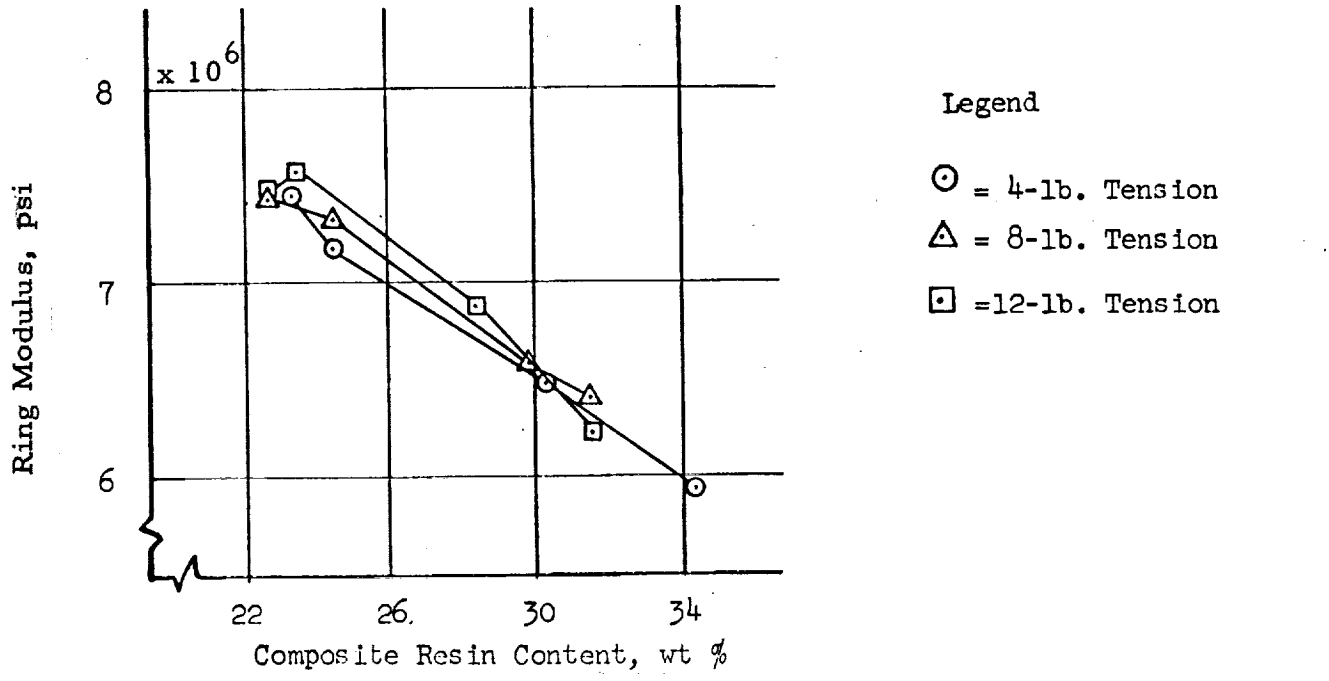


FIGURE 6: Composite Properties of Gemon-L/12-End, S-Glass NOL Rings as a Function of Resin Content

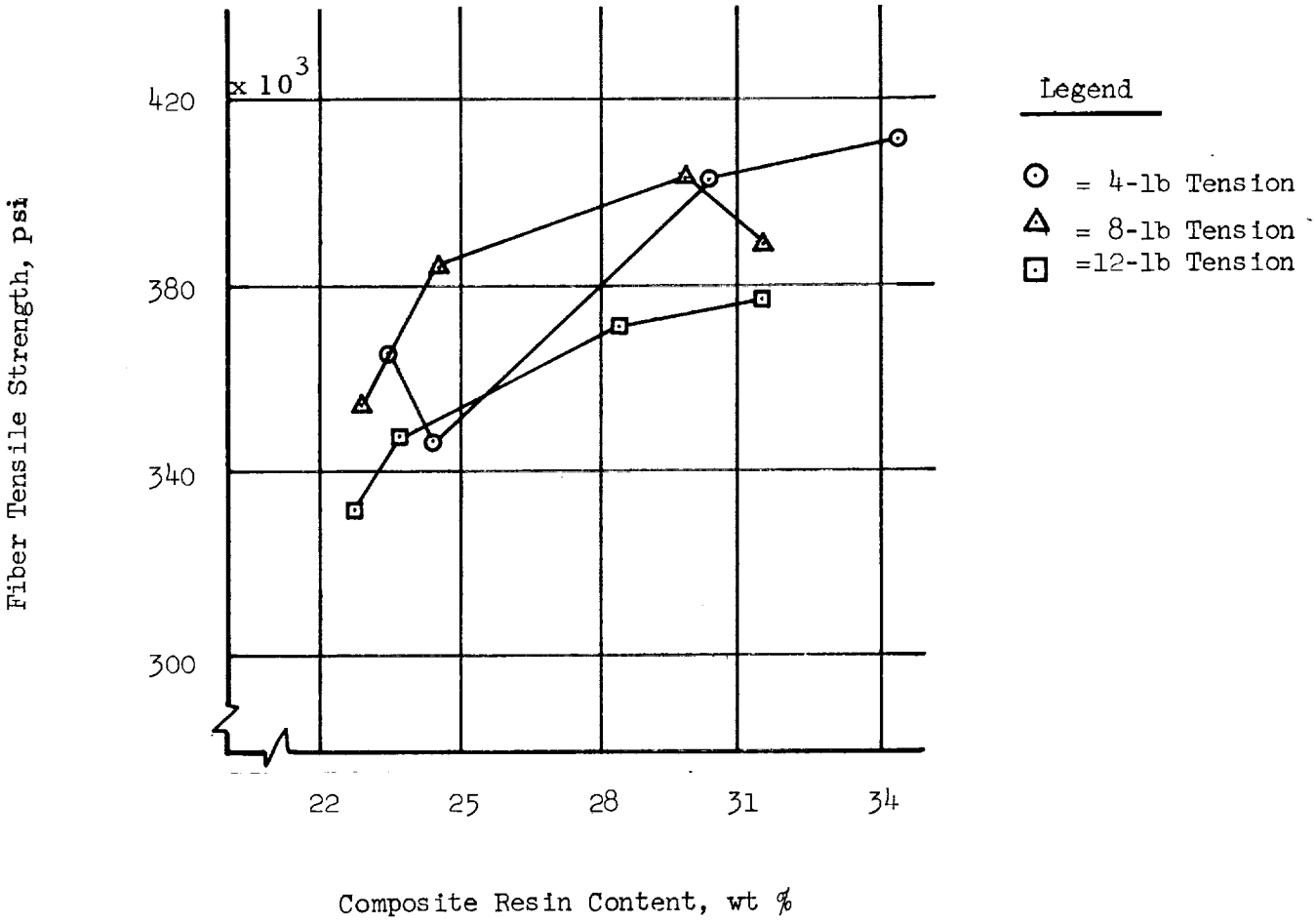
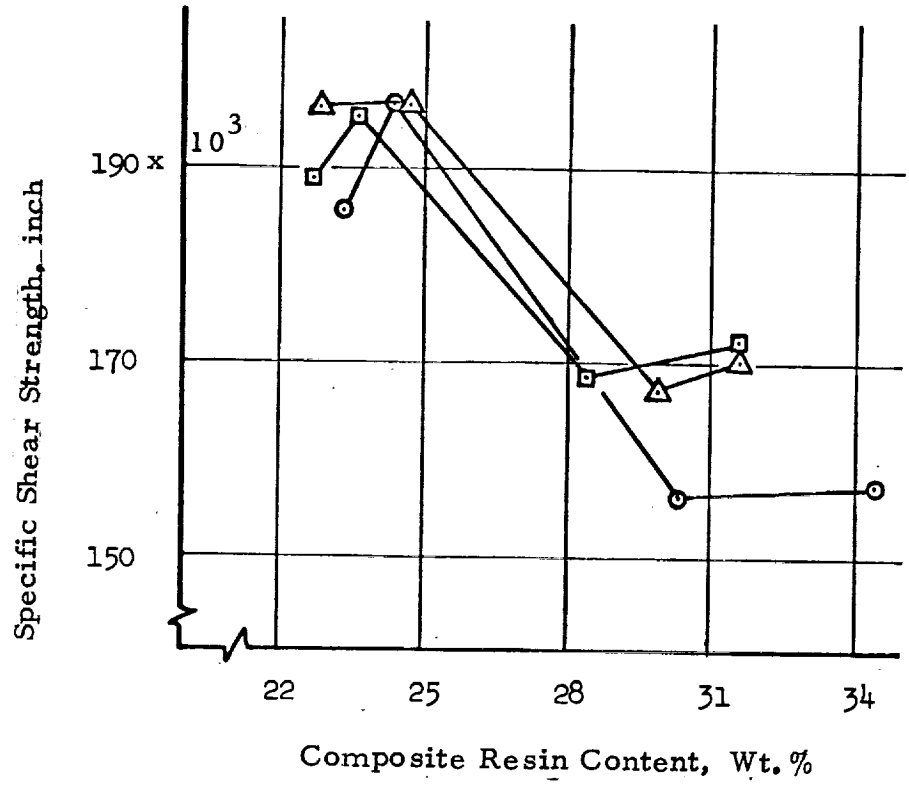


FIGURE 7: Fiber Tensile Strength of Gemon L/12-End S-Glass NOL Rings as a Function of Composite Resin Content



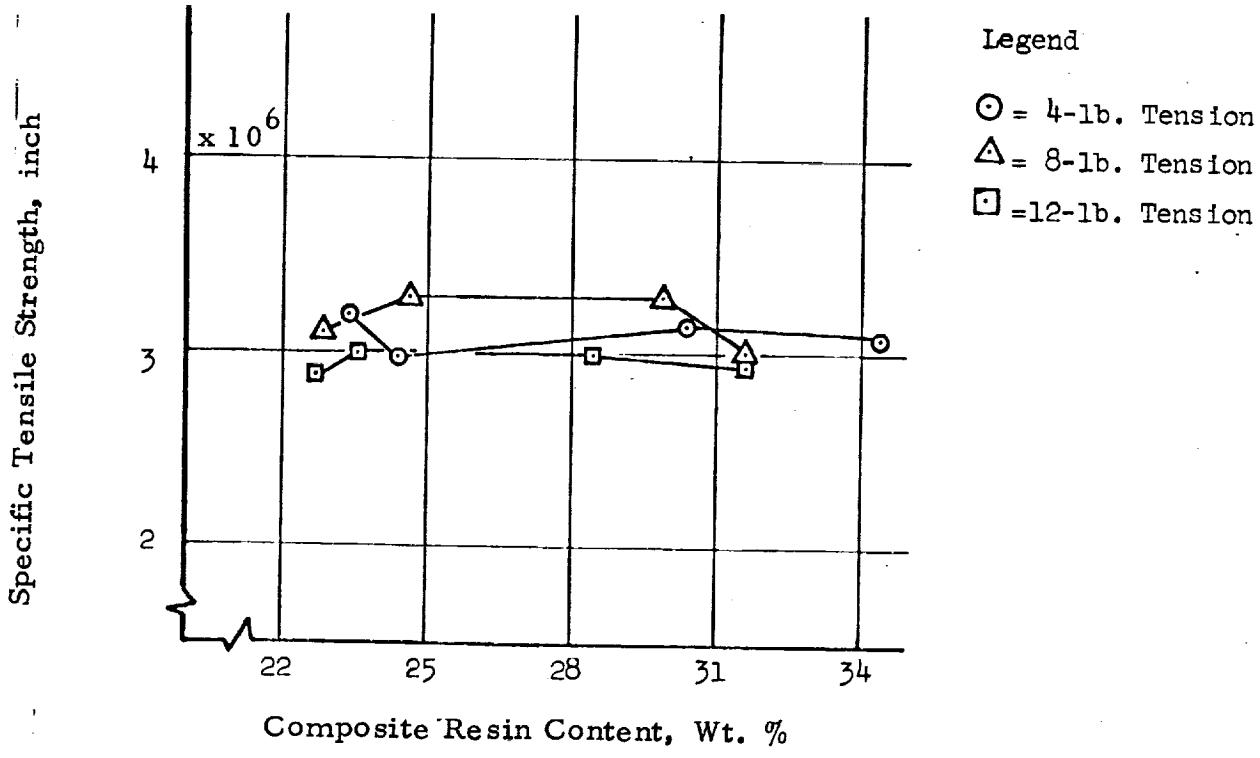
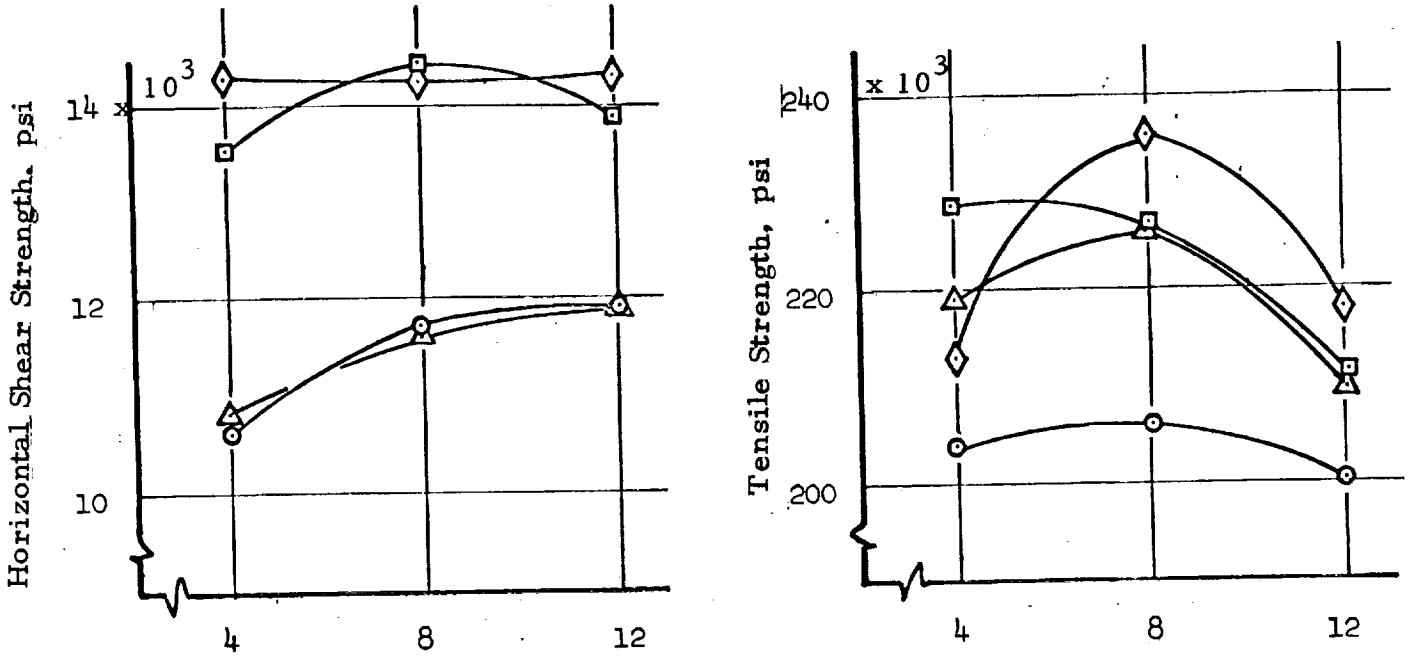


FIGURE 8: Specific Strengths of Gemon-L/12-End, S-Glass NOL Rings as a Function of Composite Resin Content



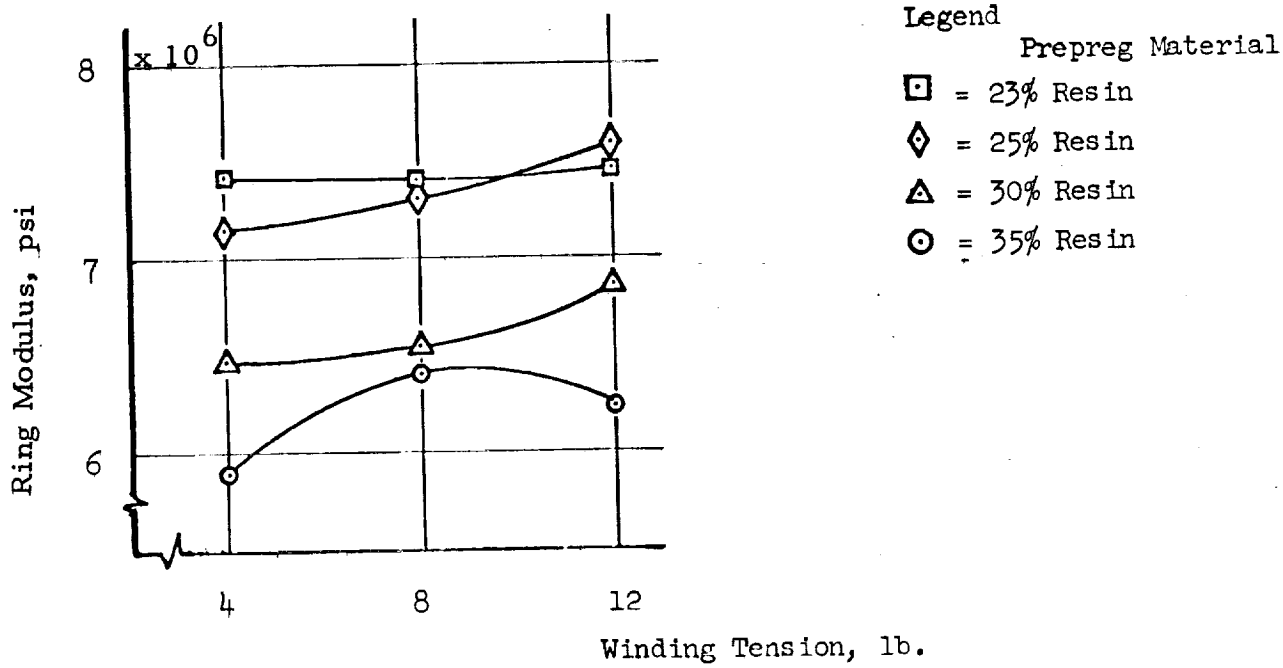
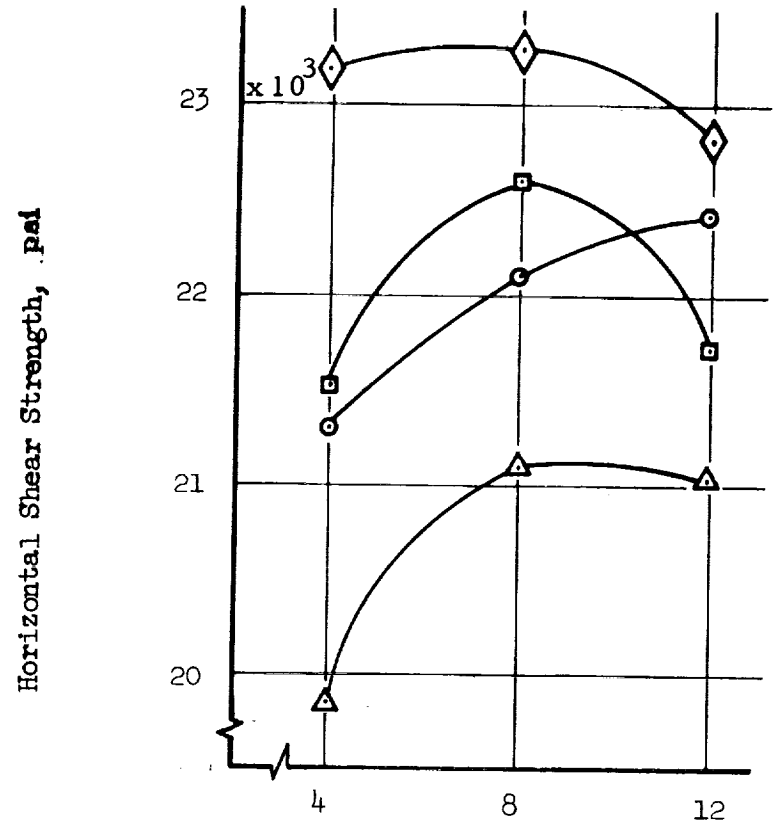


FIGURE 9: Composite Properties of Gemon L/12-End, S-Glass NOL Rings as a Function of Winding Tension



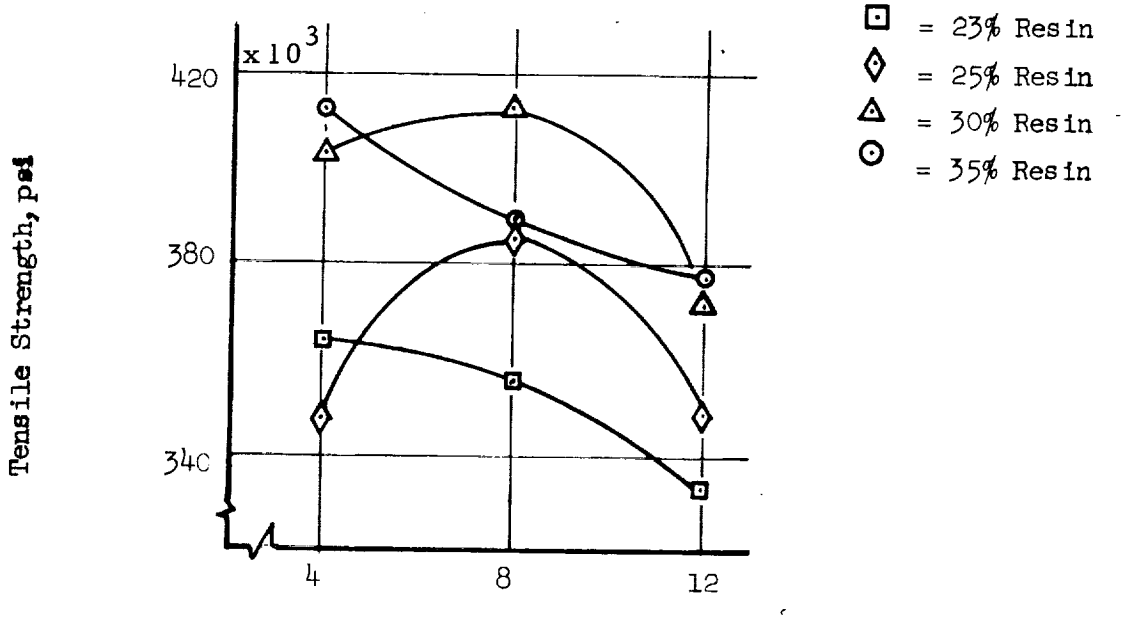


FIGURE 10: Normalized Fiber Properties of Gemon L/12-End S-Glass NOL Rings as a Function of Winding Tension

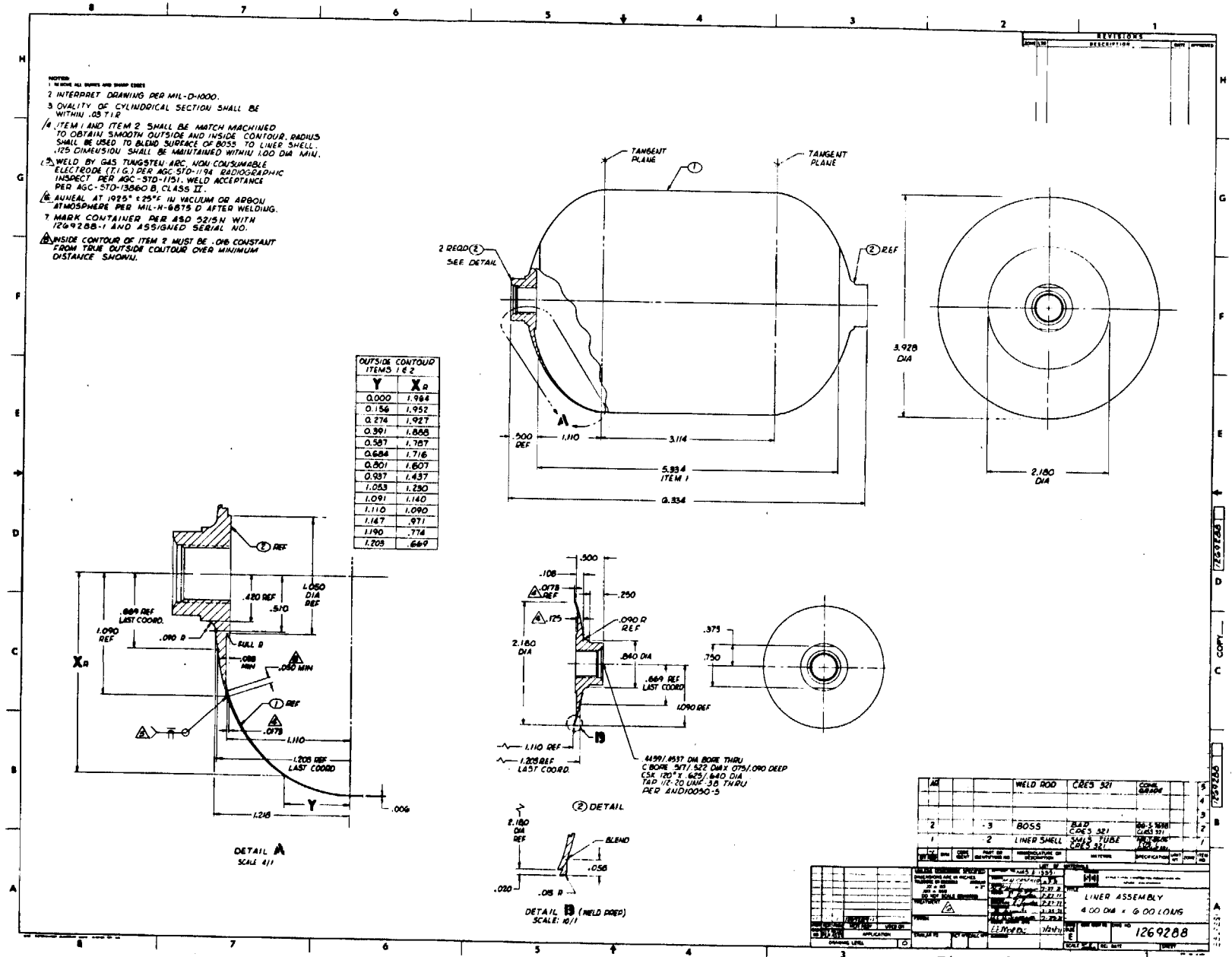


Figure 11

63-a

63-b

2. PREPARE AND OVERWRAP LINER ASSEMBLY (ITEM 1) AS SPECIFIED IN ENGINEERING MANUFACTURING INSTRUCTIONS.

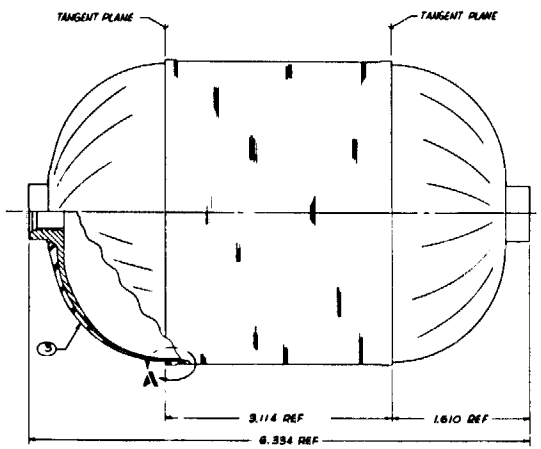
3. CLEAN OUTER SURFACE OF LINER ASSEMBLY (ITEM 1) PER AGC-STD-1221.

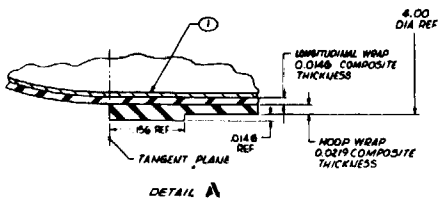
4. WINDING TENSION: 8 LBS ON 0.052 WIDE TAPE.

5. WINDING PATTERN:
   LONGITUDINAL: 1 REVOLUTION, 118 TURNS + 1 TURN FOR TIE OFF, 2 STRANDS 0.104 TAPE.
   CIRCUMFERENTIAL: 3 LAYERS, 60 TURNS PER LAYER IN 3.114 LENGTH, 1 STRAND 0.052 TAPE.
   TANGENCY BUILDUP: 2 LAYERS, 3 TURNS PER LAYER, BOTH ENDS 1 STRAND 0.052 TAPE.

6. CURING CYCLE FOR -1 ASSY ONLY:
   CURE: ROOM TEMPERATURE TO 350°F AT RATE OF 6 TO 8 °F PER MINUTE.
   2 HOURS AT 350°F
   POSTCURE: 2 HOURS AT 200°F
   2 HOURS AT 300°F
   2 HOURS AT 400°F
   16 HOURS AT 500°F

7. IN LIST OF MATERIALS 'B' IN SYMBOL COLUMN DENOTES BULK MATERIAL.





DETAIL A

REVISIONS			
ZONE	LTR	DESCRIPTION	DATE

	GLASS ROVING	12 END, S-GLASS PREIMPREGMATED WITH PIOPMA		3
	GLASS ROVING	12 END SGLASS PREIMPREGMATED WITH GEMONIL		2
1269288-1	LINER WELDED			1

PRESSURE VESSEL ASSEMBLY 4.00 DIA x 6.00 LONG

SIZE E — 1269289

1269289

Figure 12
64-b

64-a

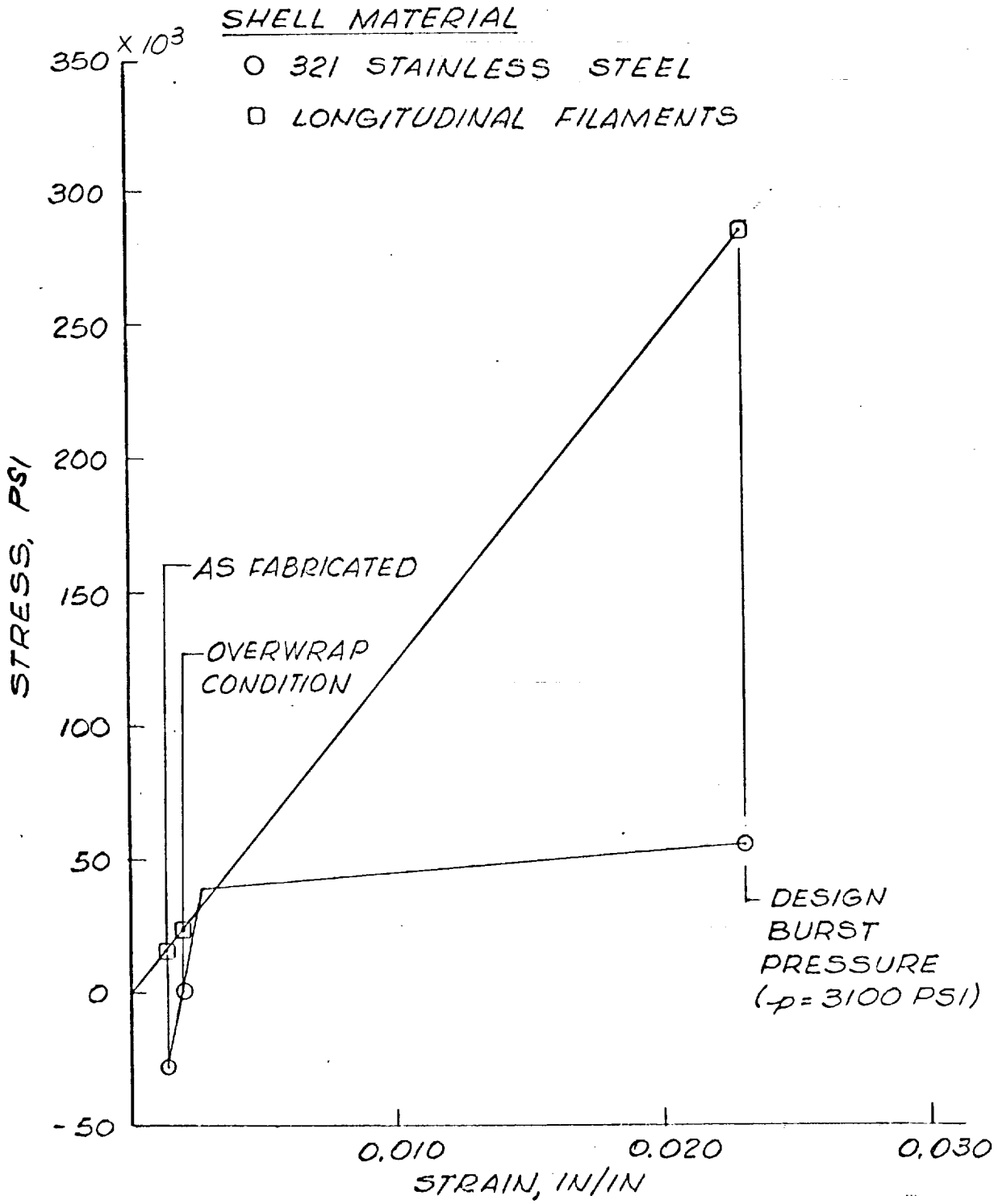


FIGURE 13
AMBIENT STRESS-STRAIN RELATIONSHIPS
LONGITUDINAL DIRECTION OF CYLINDER

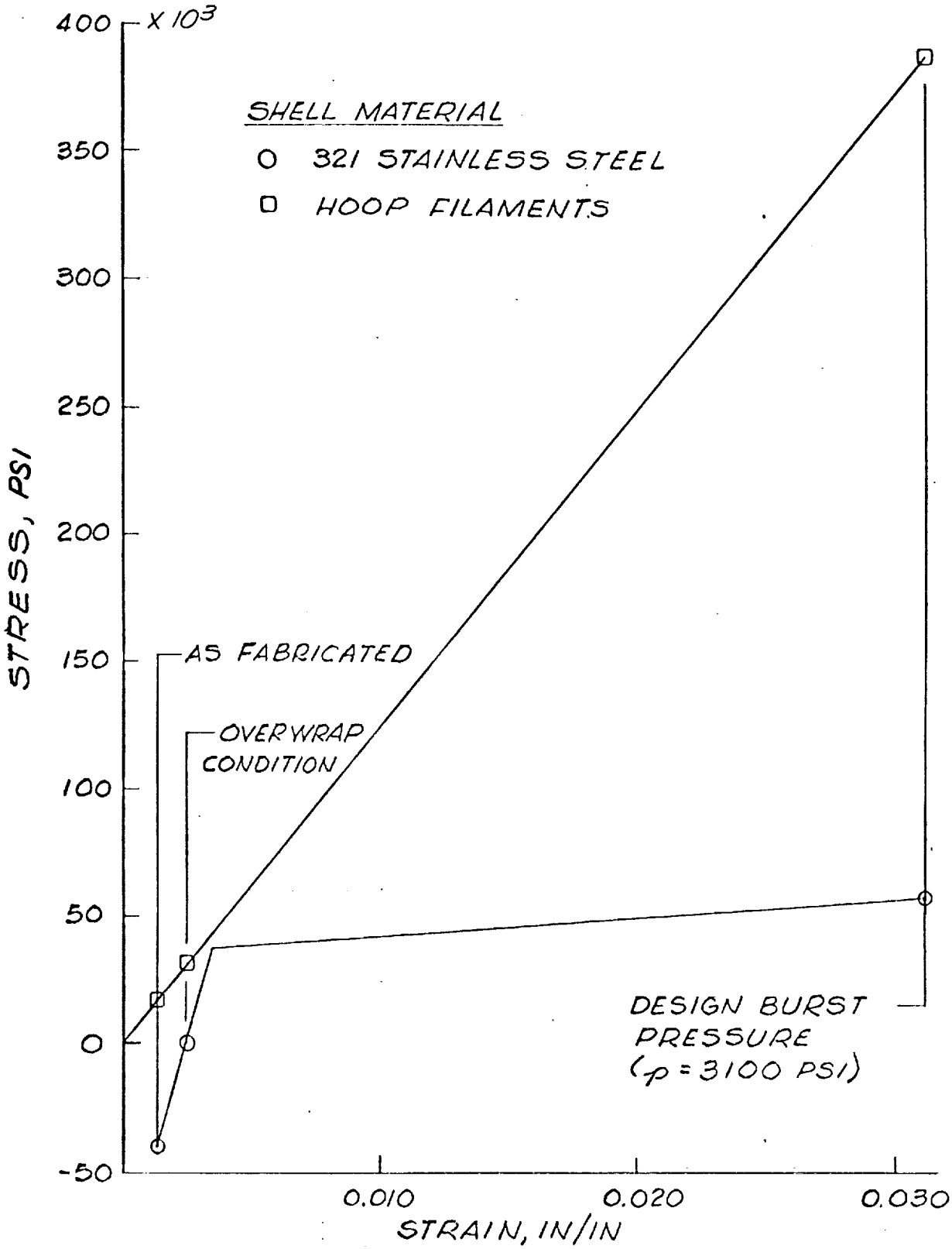


FIGURE 14
AMBIENT STRESS-STRAIN RELATIONSHIPS
HOOP DIRECTION OF CYLINDER

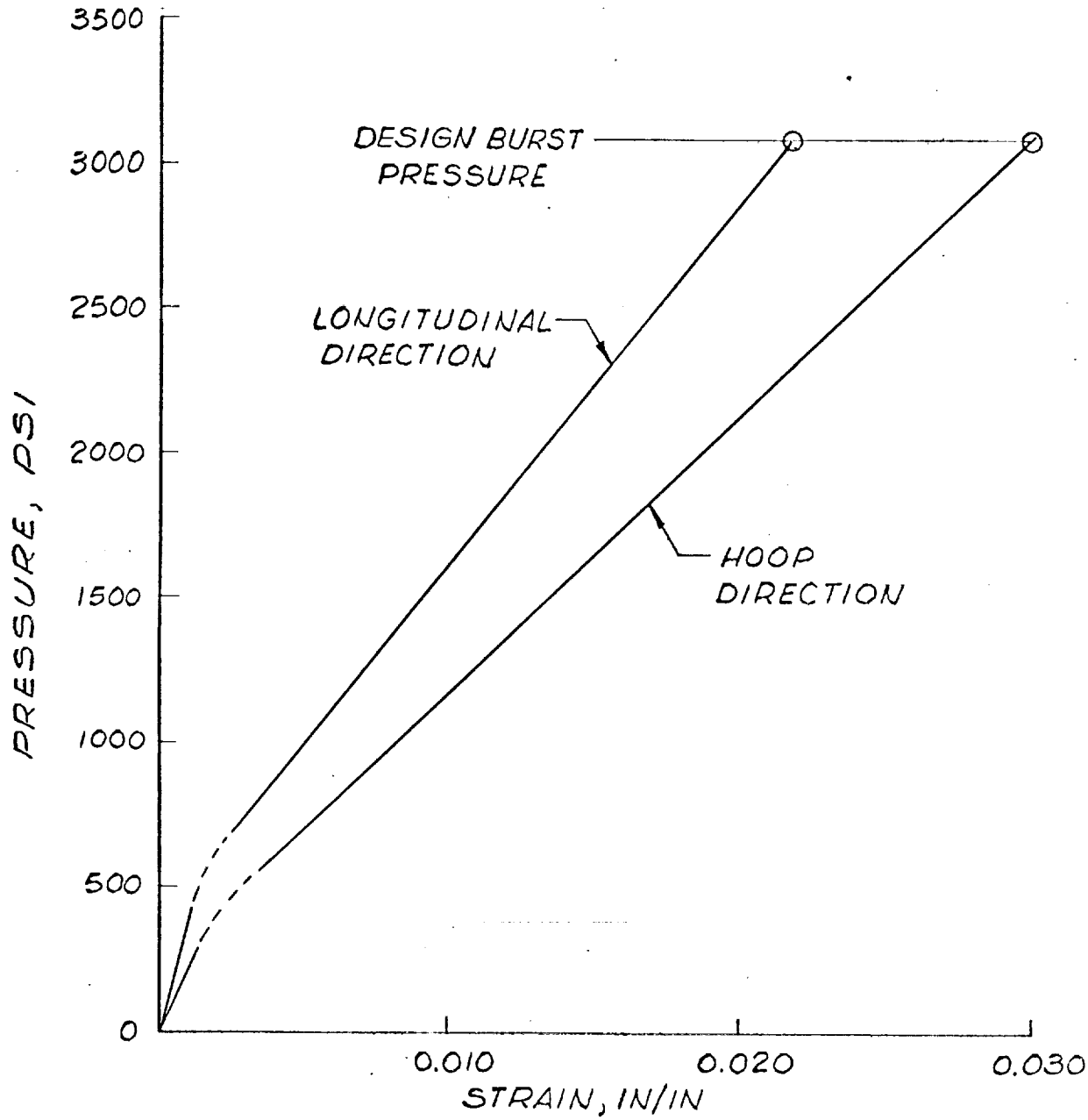


FIGURE 15

AMBIENT PRESSURE - STRAIN RELATIONSHIPS
CYLINDRICAL SECTION PRESSURE VESSEL

FIGURE 16

THIN-WALLED STAINLESS STEEL VESSEL LINERS

FIGURE 17

VESSEL FAILURE AT THE DAMAGED AREA

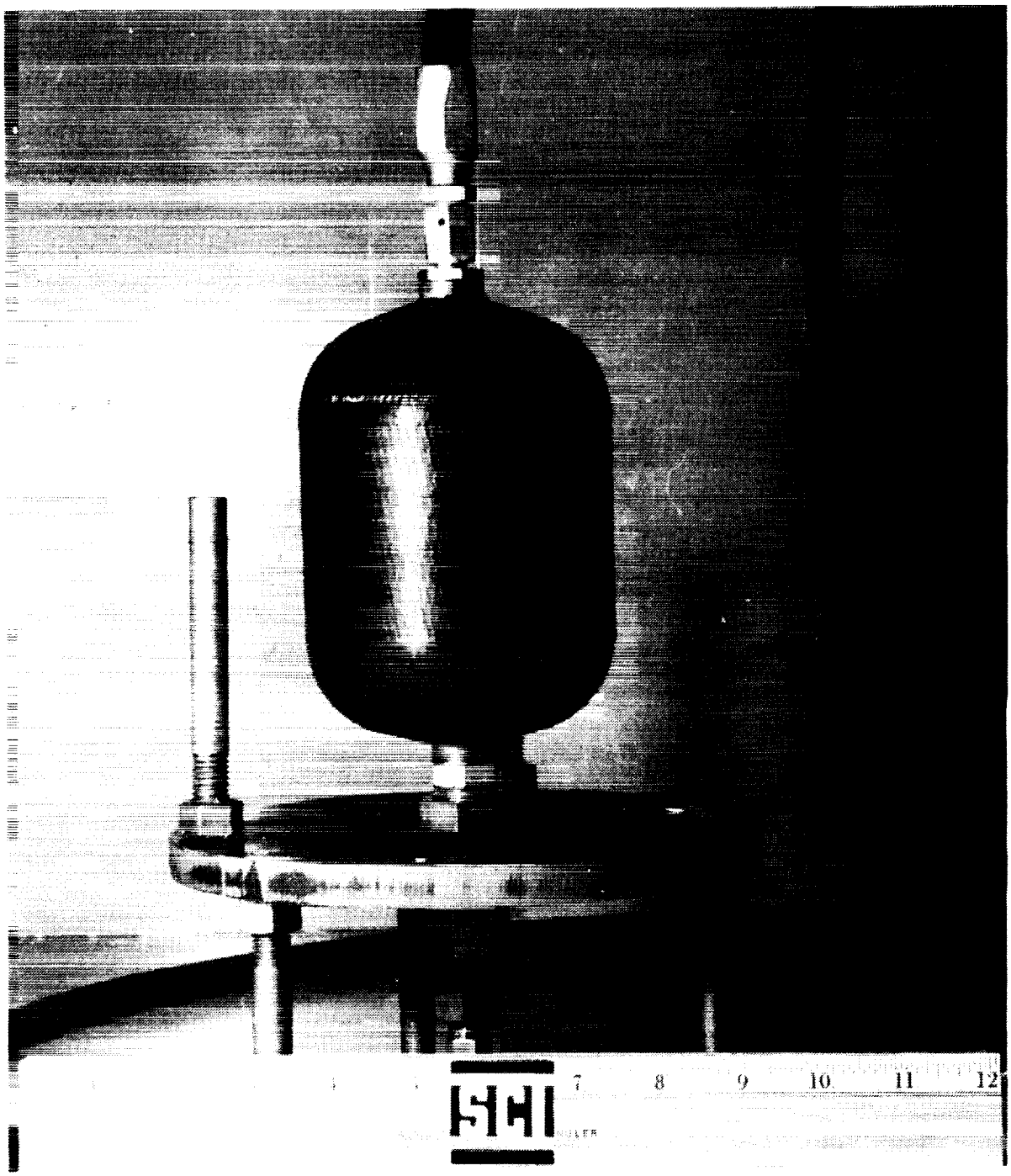


FIGURE 18

VESSEL TEST SPECIMEN BEFORE BURST TEST
S/N 1

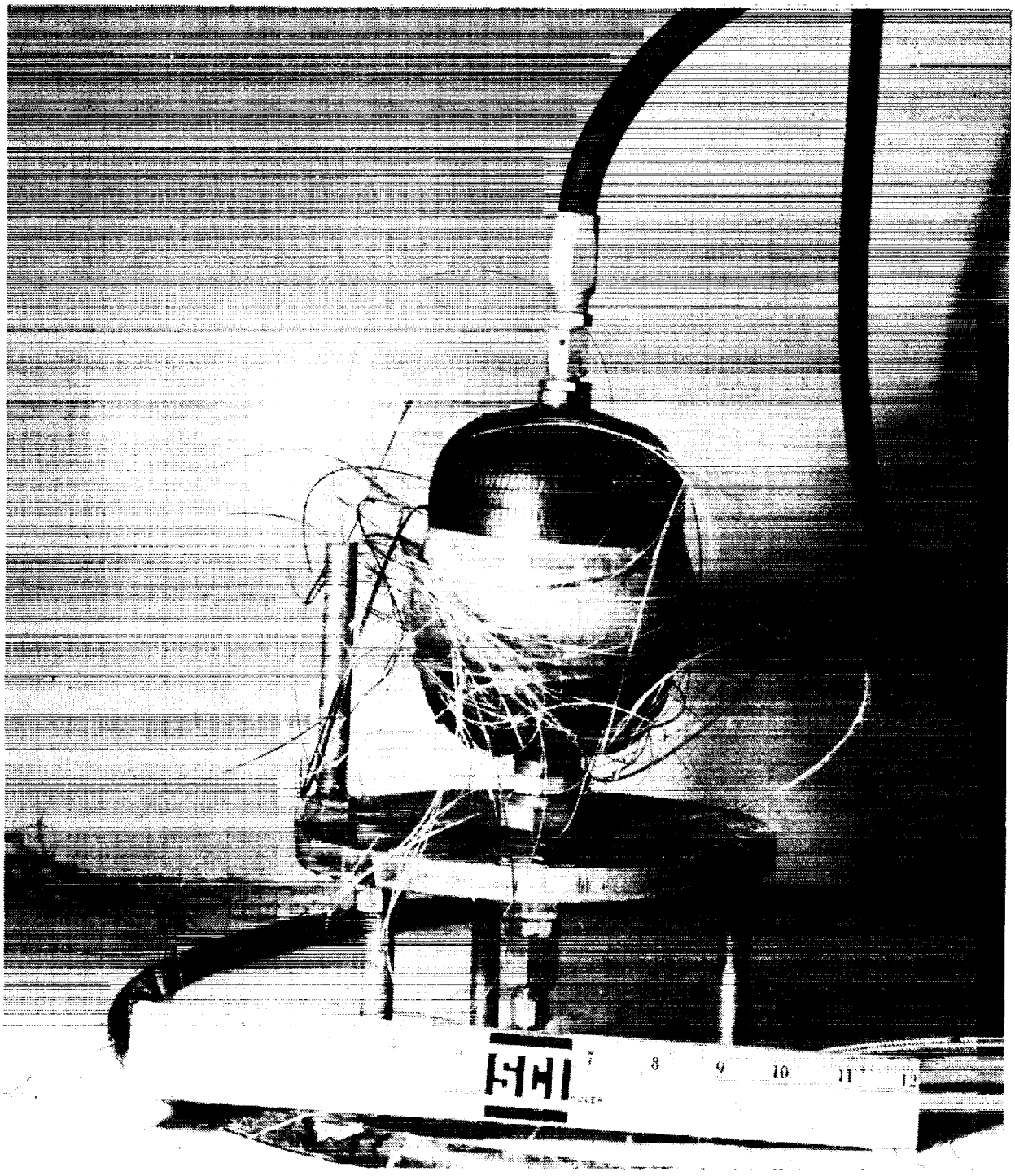


FIGURE 19

VESSEL TEST SPECIMEN AFTER BURST
TEST - SERIAL NUMBER 1

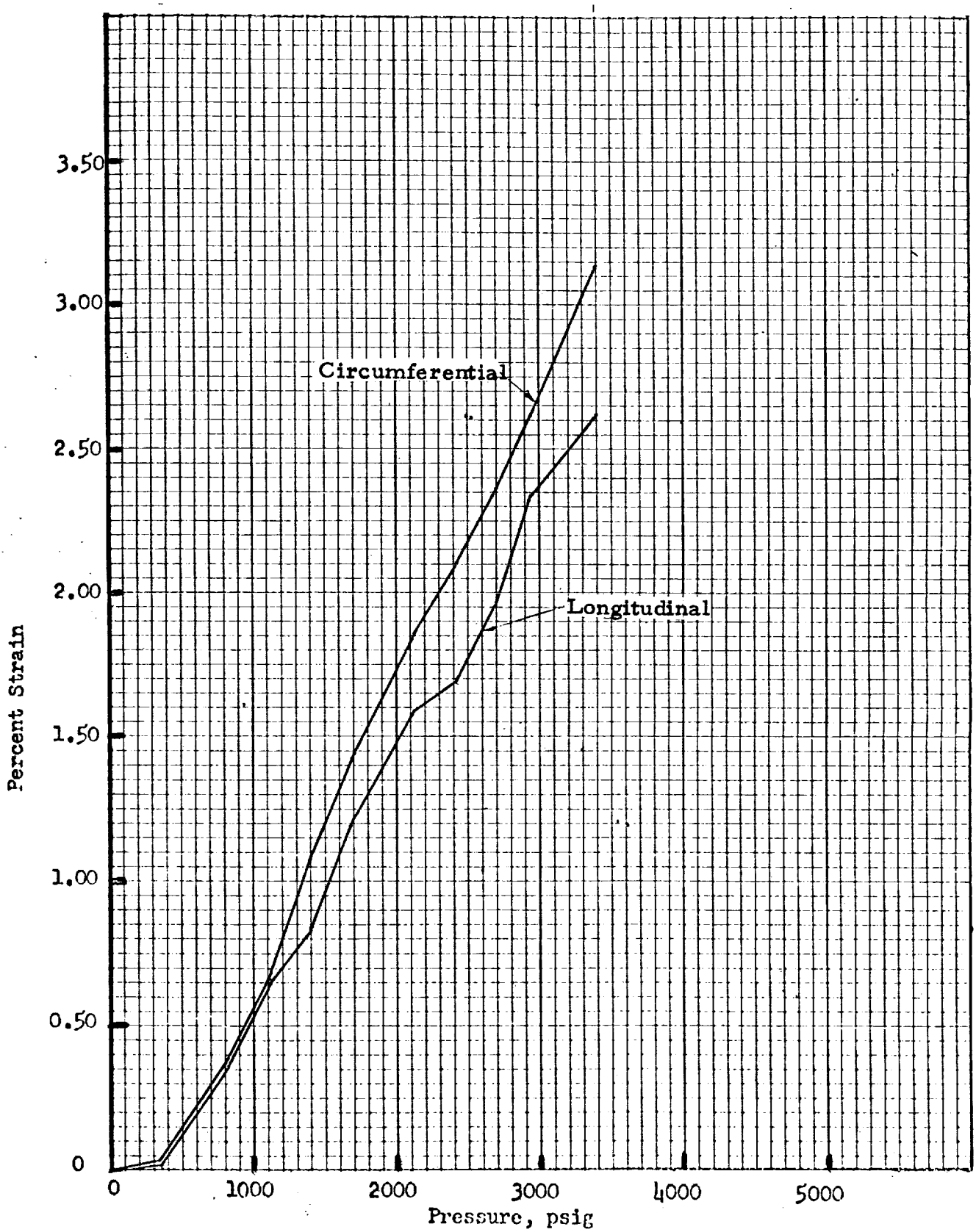


FIGURE 20
Longitudinal and Circumferential Strains as a Function of Pressure for Single-Cycle Burst Test at Ambient Temperature Vessel S/N 1

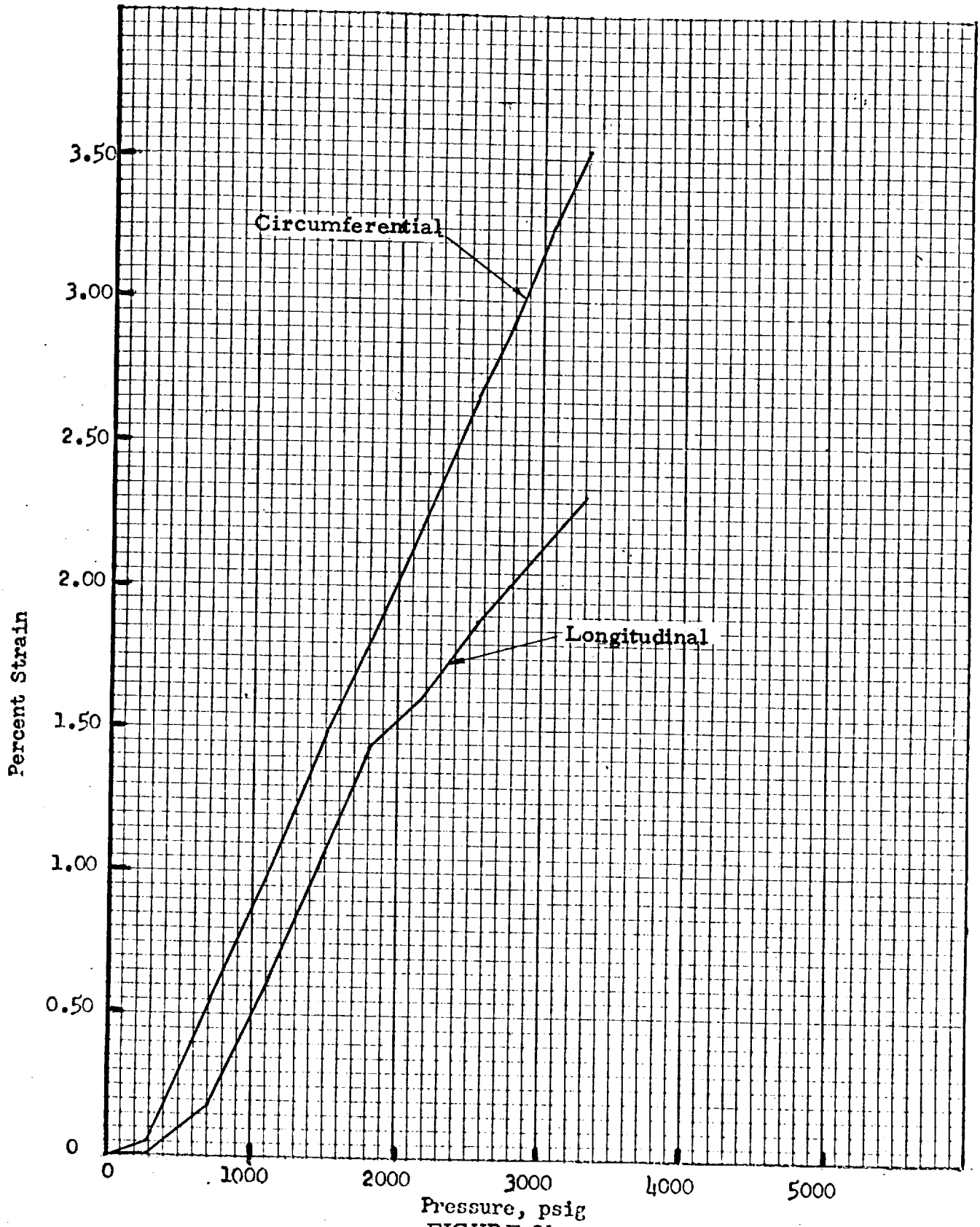


FIGURE 21

Longitudinal and Circumferential Strains as a Function of Pressure for Single-Cycle Burst Test at Ambient Temperature Vessel S/N 2

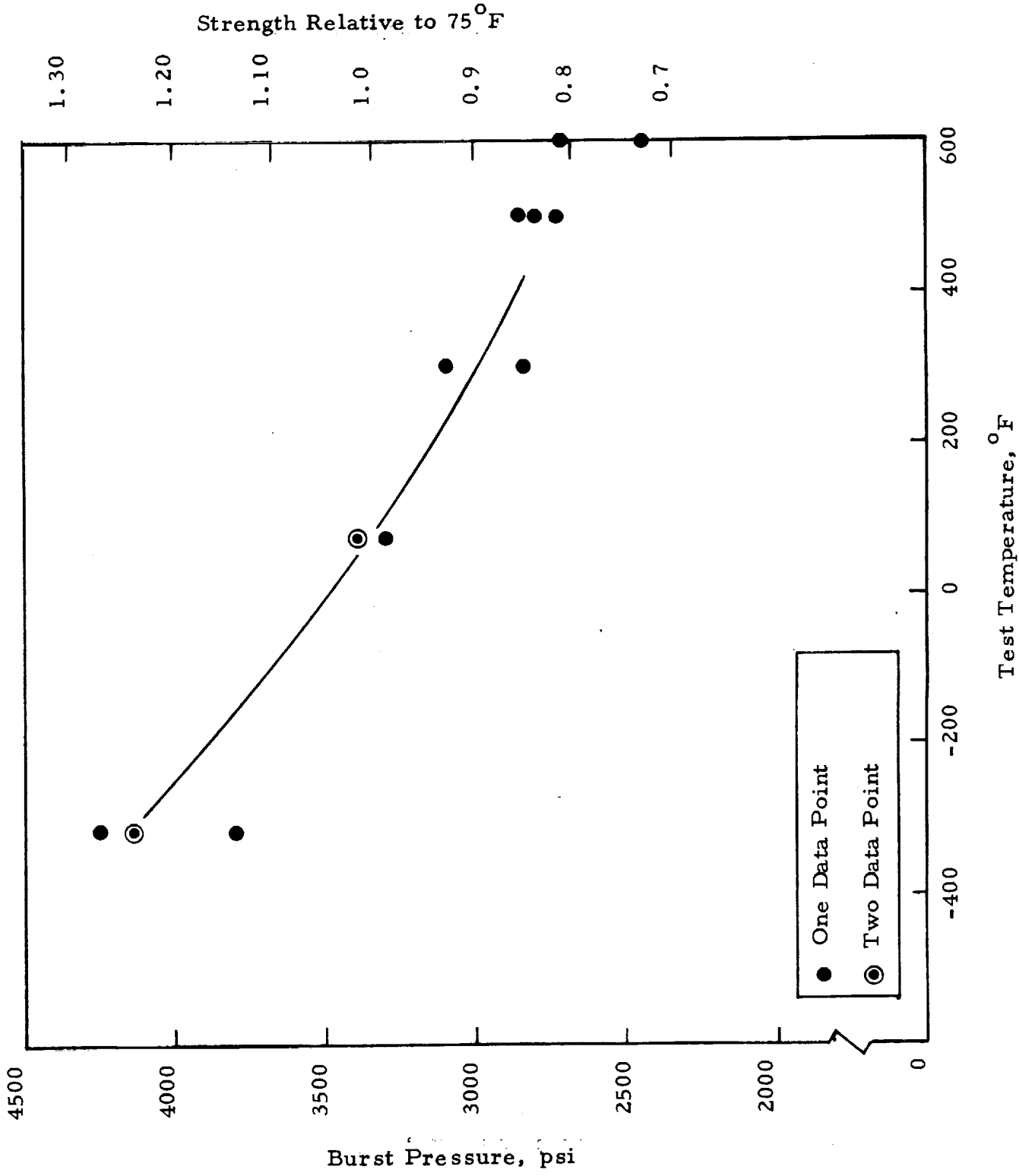


FIGURE 22

EFFECT OF TEMPERATURE ON BURST STRENGTH

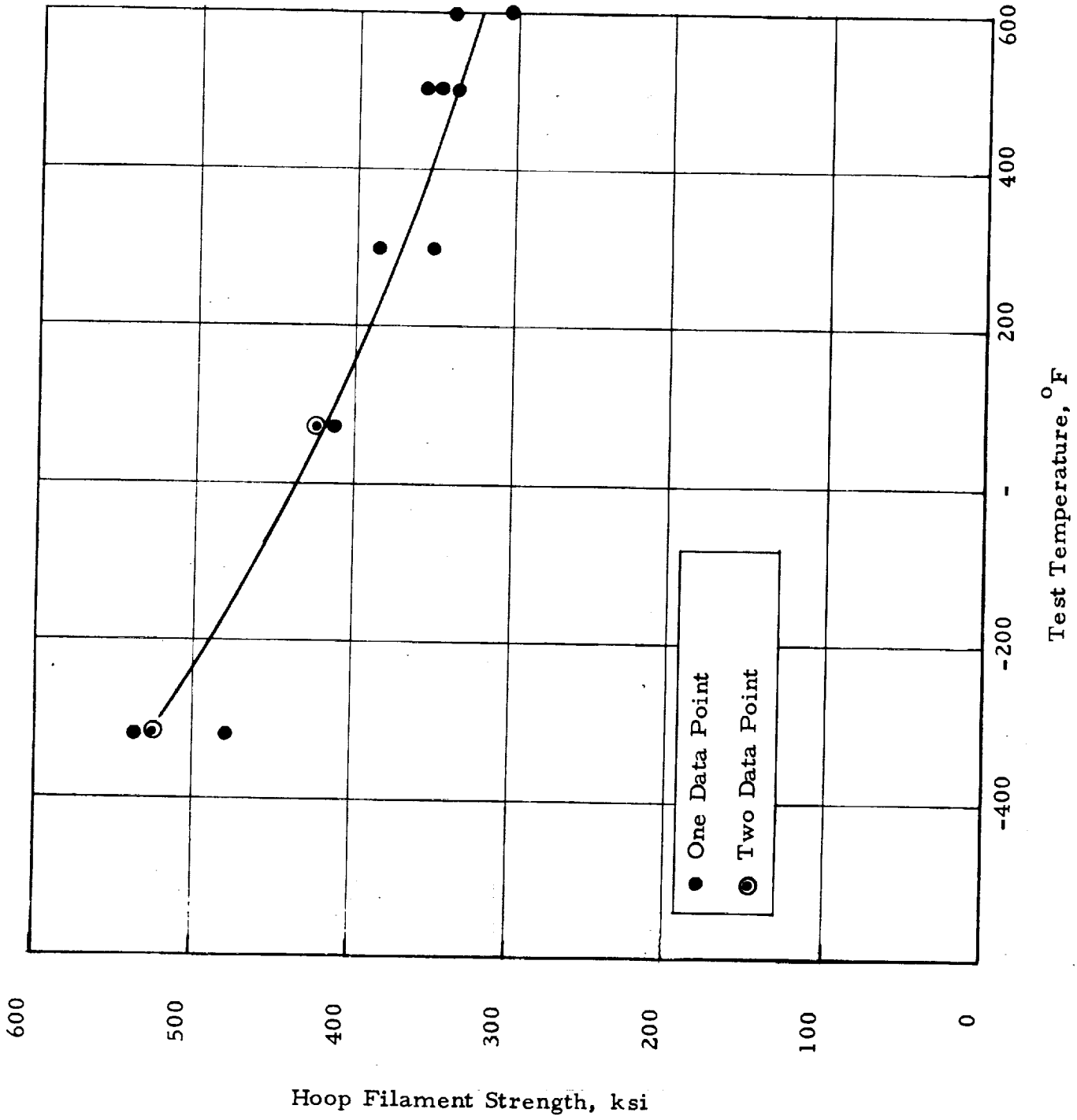


FIGURE 23

HOOP FILAMENT STRENGTH AS A FUNCTION OF TEMPERATURE

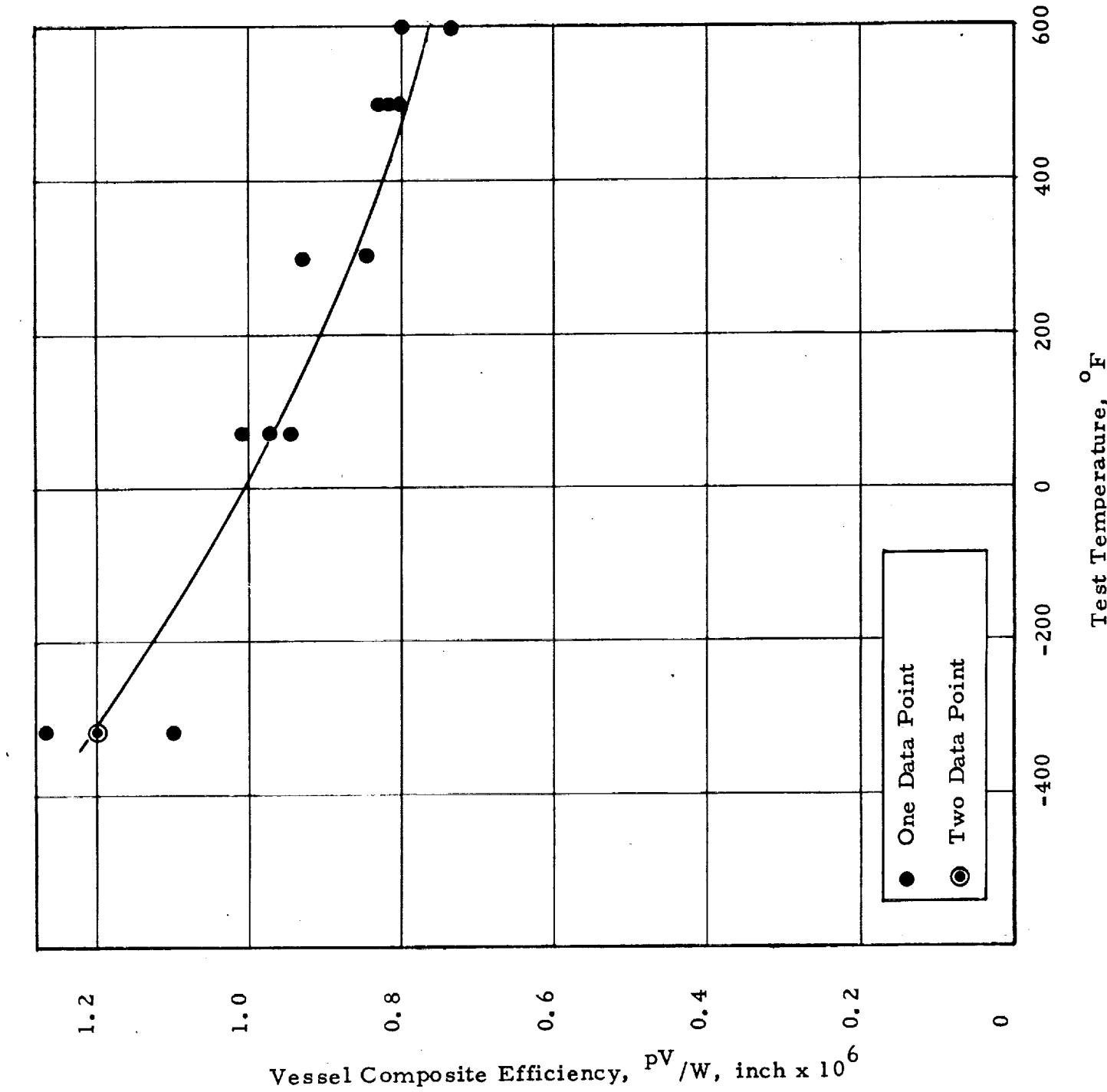


FIGURE 24

VESSEL COMPOSITE EFFICIENCY ($^{pV}/_{W}$) AS A FUNCTION OF TEMPERATURE

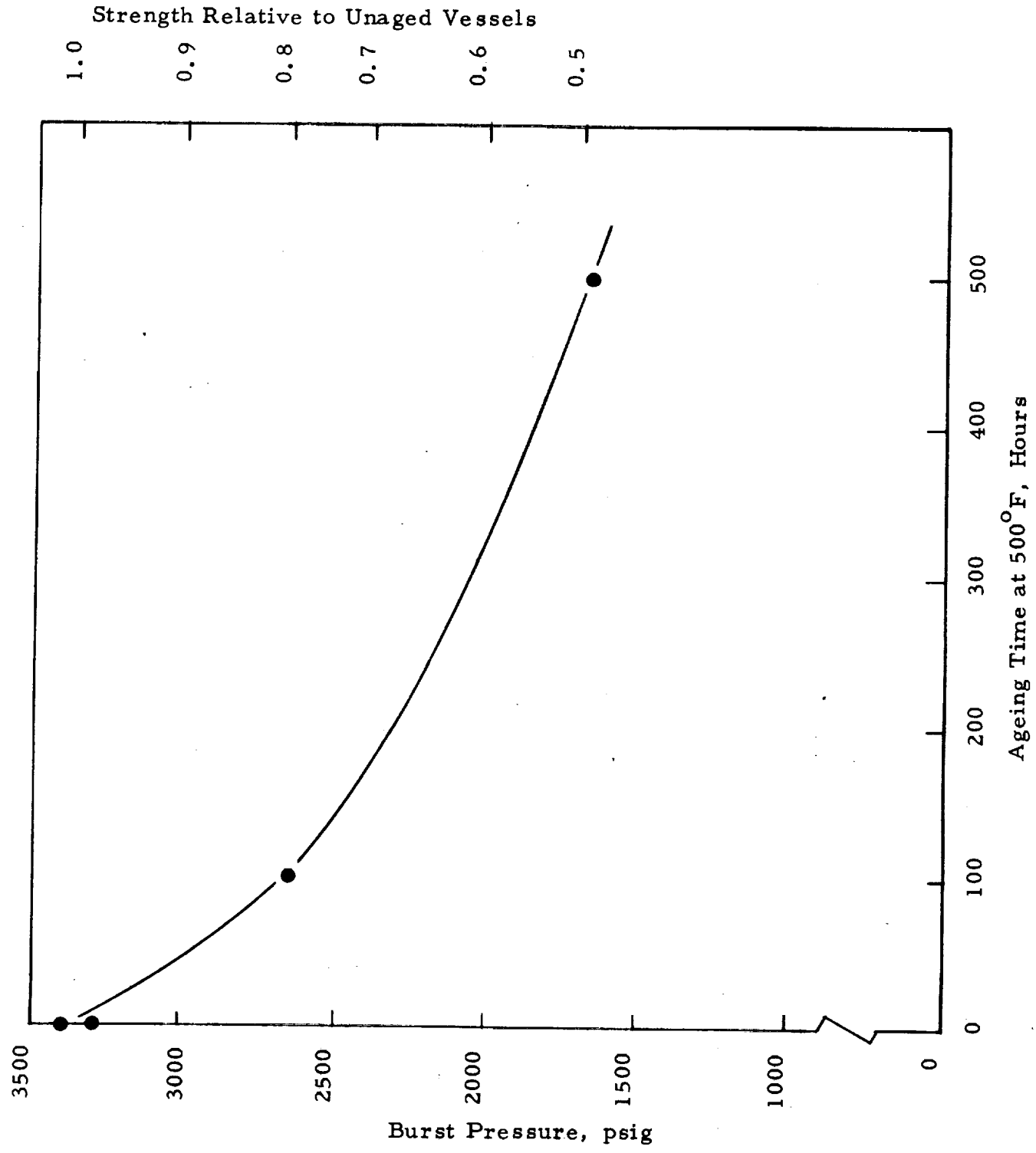


FIGURE 25

EFFECT OF THERMAL AGEING ON BURST STRENGTH

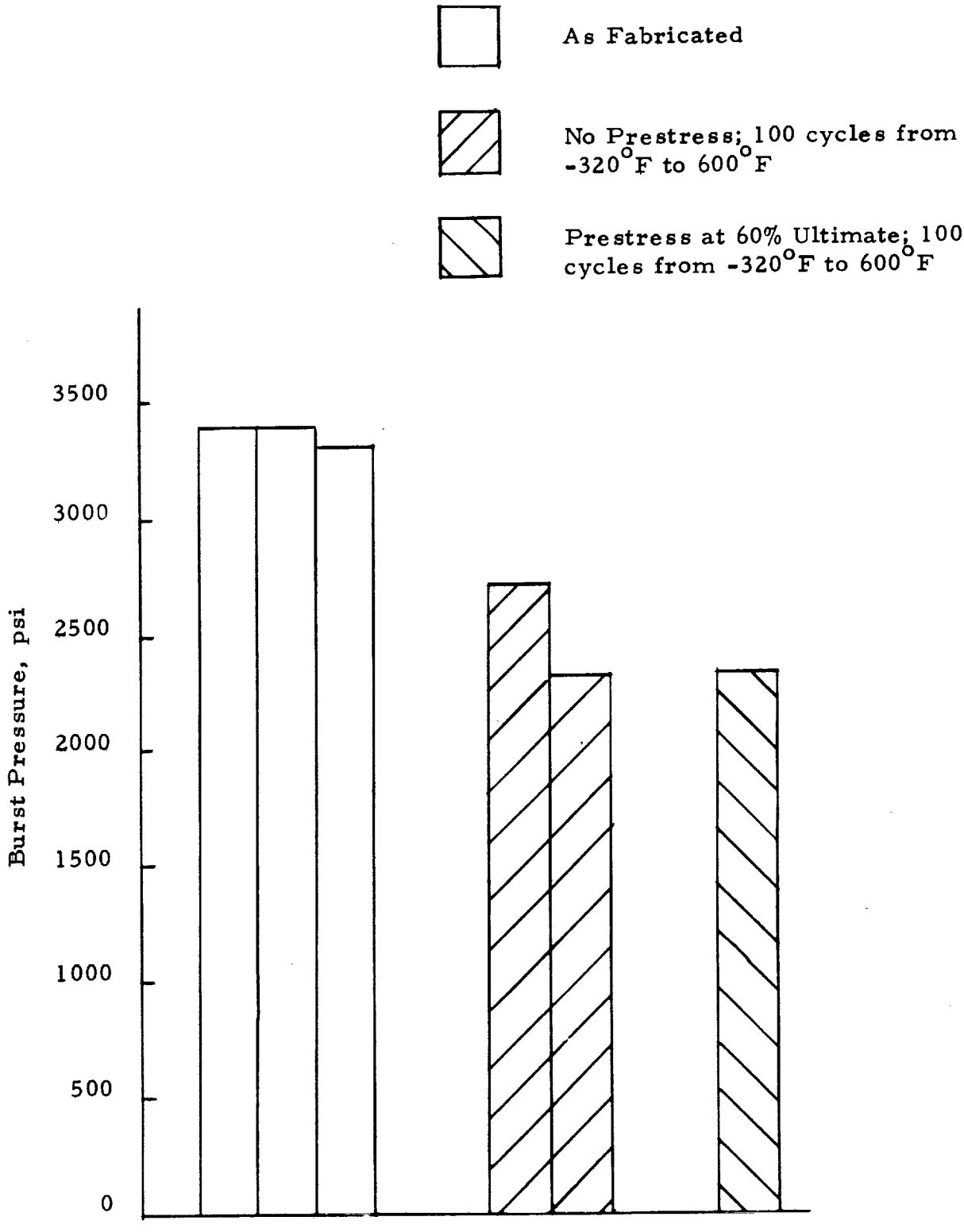


FIGURE 26

EFFECT OF THERMAL CYCLING ON BURST STRENGTH

STRUCTURAL COMPOSITES INDUSTRIES INC.
6344 NORTH IRWINDALE AVENUE AZUSA, CALIFORNIA 91702 (213) 334-8221

APPENDIX A

DESIGN ANALYSIS OF 4-INCH-DIAMETER BY 6-INCH-LONG 321 STAINLESS STEEL LINED GLASS FILAMENT WOUND/POLYIMIDE RESIN COMPOSITE PRESSURE VESSEL

DESIGN ANALYSIS OF 4-IN.-DIA BY 6-IN.-Long 321 SS-LINED GLASS FILAMENT-WOUND/POLYIMIDE RESIN COMPOSITE PRESSURE VESSEL

CONTRACT NAS 3-15551

Prepared for

NASA-LEWIS RESEARCH CENTER

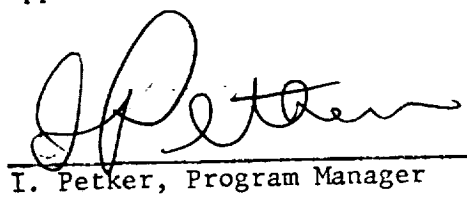
CLEVELAND, OHIO

Prepared by

R. E. LANDES

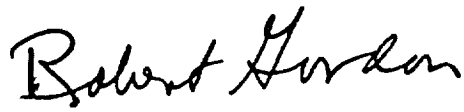
July 1971

Approved:



I. Petker, Program Manager

Approved for distribution:



Robert Gordon, President

STRUCTURAL COMPOSITES INDUSTRIES, INC.

6344 N. Irwindale Ave.

Azusa, California

DESIGN ANALYSIS
GLASS FILAMENT-WOUND/POLYIMIDE
RESIN COMPOSITE PRESSURE VESSEL

This report covers the design and analysis of a 4-in.-diameter by 6-in.-long cylindrical closed-end, metal-lined filament-wound pressure vessel to be used as a test specimen for evaluation of biaxially loaded Glass/Polyimide composites.

I. DESIGN CRITERIA

Fiber/Matrix - Twelve-end S-Glass filaments in continuous length and preimpregnated with General Electric polyimide resin (Gemon L), and TRW's P-10PMA polyimide resin.

Shape - Closed-end cylinder

Size - 4-in.-diameter by 6-in.-long

Liner - Stainless Steel, Type 321 (Annealed), 0.006-in. thickness

Winding Pattern - In plane

Winding Tension - 6 lb./12-end strand*

Fiber Content - 67 volume percent*

Service Temperatures - 75°F, 600°F, -423°F

Burst Pressure - Dictated by minimum wrap thicknesses

Reference Drawings:

Pressure Vessel - SCI Drawing No. 1269288

Liner Assembly - SCI Drawing No. 1269289

* Preliminary values; actuals to be determined during Program Task I work.

II. DESIGN ALLOWABLE GLASS-FILAMENT STRENGTH

Aerojet/SCI has developed a systematic approach to the design of filament-wound vessels (Reference 1, 2, and 3) and is using it in a number of applications. The method involves the use of pressure-vessel design factors, corresponding to a range of dimensional parameters, to determine the allowable strength for each configuration. The factors are based on data collected over the past 10 years from tests on several thousands of pressure vessels; these vessels ranged in diameter from 4 to 74 in. and had significant variations in their design parameters. Included as factors used for the selection of design-allowable values are the strength of the glass roving, resin content, envelope dimensions (length and diameter), internal pressure level, axial port diameters, temperature, sustained loading requirements, and cyclic loading requirements. The method was used in this analysis to establish realistic values for the allowable ultimate 75°F S-glass-filament tensile strengths in the 4-in.-dia. by 6-in.-long stainless steel lined filament-wound test vessel.

A. LONGITUDINAL FILAMENTS

The allowable longitudinal-filament strength is given by

$$F_{f,l} = K_1 K_2 K_3 K_4 K_5 (\sec^2 \alpha) F_f$$

The following design factors (Reference 3) are based on the specific vessel parameters:

Parameter	Design Factor
$D_c = 3.942$	0.900 (K_1)
$D_b/D_c = 0.21$	0.995 (K_2)
$L/D_c = 1.4$	0.995 (K_3)
$t_{f,l}/D_c \approx 0.0015$	0.960 (K_4)
$T = 75^\circ F$	1.000 (K_5)
$\alpha = 10.3^\circ$ (from geometry of vessel)	

For S-glass filaments, the minimum ultimate tensile strength, F_f, is 415,000 psi.

The single-pressure-cycle allowable ultimate longitudinal filament strength is therefore

$$F_{f,l} = (0.900)\ (0.995)\ (0.995)\ (0.960)\ (1.000)\ (1.032)\ (415{,}000)$$

$$= 366{,}000 \text{ psi}$$

B. HOOP FILAMENTS

The allowable hoop-filament strength is given by

$$F_{f,h} = K_1 K_4 K_5 \left(1 - \frac{\tan^2 \alpha}{2}\right) F_f$$

The following design factors are based on the specific vessel parameters

Parameter	Design Factor
D_c = 3.942 in.	0.964 (K_1)
$t_{f,h}/D_c$ 0.00225	0.990 (K_4)
T = 75°F	1.000 (K_5)
α = 10.3°	

The single-pressure-cycle allowable ultimate hoop filament strength is therefore

$$F_{f,h} = (0.964)\ (0.990)\ (1.000)\left[1-\frac{0.03313}{2}\right]415{,}000$$

$$= 390{,}000 \text{ psi}$$

III. WINDING PATTERN ANALYSIS

The filament-wound vessel has two winding patterns: a longitudinal-in-plane pattern along the cylinder and over the end domes to provide the total filament-wound composite strength in the heads and the longitudinal strength in the cylindrical section; and a circumferential pattern applied along the cylinder for hoop strength in this section.

The winding pattern for the pressure vessel requires the application of a specific quantity of glass roving in predetermined orientations in order to obtain the desired burst pressure. The filament thickness per layer of 12-end S-glass/PI prepreg used in vessel winding may be determined from the expression

$$t_f = A_f/W_f$$

Where

A_f = cross sectional area of 12-end roving

$= 2.535 \times 10^{-4}$ in^2

W_f = single strand tape width, fixed at 0.052 in.

Therefore, for both longitudinal and hoop patterns, the filament thickness per layer is

$$t_f = 2.535 \times 10^{-4}/.052 = 0.00488 \text{ in.}$$

A. LONGITUDINAL PATTERN

Two layers are formed for each revolution (N) of the winding mandrel. Since the vessel is to be minimum burst pressure unit, the number of revolutions was fixed at one, which establishes the number of longitudinal layers (N_l) at two. The resulting thickness of the longitudinal composite is calculated from the expression

$$t_l = N_l\ t_f/P_{vg}$$

where, the volume fraction of glass (P_{vg}) was preliminarily selected as 0.67. Thus,

$$t_1 = 2(.00488)/.67 = 0.0146 \text{ in.}$$

The winding tape width (W_1) is given by the expression

$$W_1 = N_2 W_f$$

Where, the number of 12-end strands (N_2) was selected as two. Thus,

$$W_1 = 2(.052) = 0.104 \text{ in.}$$

The number of turns per revolution (N_3) must be an integer, and is calculated from the relation

$$N_3 = \frac{\pi D_c \cos \alpha}{W_1 + \epsilon_{tp}}$$

where,

ϵ_{tp} = space between tapes (which should equal zero)

D_c = Vessel neutral axis diameter = $D_o - 2t_h - t_1$

and

D_o = vessel outside diameter = 4.000 in.

For a hoop composite thickness (t_h) of 0.0219 in. (see sections III-B and IV-B)

$$D_c = 4.000 - 2(.0219) - .0146 = 3.942 \text{ in.}$$

and

$$N_3 = \frac{\pi (3.942)\ (.9868)}{0.104} = 118 \text{ turns per revolution}$$

B. HOOP PATTERN

The required number of layers of hoop winding (N_h) to force failure in the cylindrical section was selected as three (see section IV of this analysis). The corresponding hoop composite thickness (t_h) is calculated from the expression

$$t_h = N_h f / P_{vg}$$

and, using previously defined values for the variables

$$t_h = 3(0.00488)/0.67 = 0.0219 \text{ in.}$$

The number of turns per inch of cylinder length (N_5) is given by the expression

$$N_5 = L_c / N_4 W_f$$

Where,

L_c = cylinder length, selected as 3.114 in.

N_4 = number of 12-end strands per tape, selected as 1

Therefore,

$N_5 = 3.114/(1)(0.052) = 60$ turns per inch (per layer)

The preceding winding pattern details have been made a part of the pressure vessel drawing No. 1269288.

IV. MEMBRANE ANALYSIS

A. METHOD

The vessel shape and burst pressure were established with a previously developed computer program for analysis of metal-lined filament-wound pressure vessels (Reference 4). The program was used to investigate the filament shell by means of a netting analysis, which assumes constant stresses along the filament path and that the resin matrix makes a negligible structural contribution. The filament and metal shells are combined by equating strains in the longitudinal and hoop directions and by adjusting the shell radii of curvature to match the combined material strengths at the design pressure.

The program established the optimum head contour and other dimensional coordinates, as well as the shell stresses and strains at zero pressure and the design pressure, the filament-path length, and the weight and volume of the components and complete vessel. It was also used to determine the stresses and strains in the two shells during vessel operation through the use of a series of pressures and temperatures.

B. COMPUTER INPUT AND OUTPUT

As previously stated in Section III-A, the number of longitudinal composite layers was minimized to achieve a minimum burst pressure. Selection of the number of hoop layers was based on the additional condition that vessel failure occur in the hoop fibers of the cylindrical section. This unbalanced* design condition is desirable to ensure reproducibility of vessel burst test data and failure modes. Section II of this report indicated that a balanced design would have an ultimate longitudinal-to-hoop strength ratio of 0.94; preliminary analysis indicated four hoop layers would produce a stress ratio of 0.98 (balanced design), whereas three hoop layers would produce a stress ratio of 0.74. Thus, to ensure a hoop filament failure, the three hoop layer system was selected to develop the 390,000 psi allowable hoop filament stress. The corresponding computer input stress level for the longitudinal filaments was 287,500 psi.

---

* A balanced design produces hoop and longitudinal filament stresses which are both at their respective design ultimate strengths at the design burst pressure; failure can occur in the cylinder, at the head knuckle, at the port, or any combination of these areas.

Additional computer input variables used to establish the vessel design are listed in Table 1. Computer output described the pressure vessel membrane contour and thicknesses, component weights, and stress/strain conditions, and established a design burst pressure of 3100 psi at 75°F. The geometric output is depicted in the Reference Drawing Numbers 1269288 and 1269289.

Computer derived ambient stress-strain relationships for the longitudinal direction of the cylinder in the filaments and the liner are shown in Figure 1 and for the hoop direction in Figure 2 - up to the theoretical burst pressure. Computer output was also used to construct pressure-strain curves for the ambient test condition. These curves, presented in Figure 3, will be used later in the program to compare the measured pressure-strain characteristics of vessels with the predicted behavior.

V. BOSS DESIGN ANALYSIS

A. CONFIGURATION

The metal boss is fabricated from annealed type 321 Stainless Steel. The significant dimensions of the boss used for this analysis were taken from the Reference Drawing Number 1269289.

B. MATERIAL PROPERTIES

Type 321 Stainless Steel (annealed) has the following minimum strength properties

	Strength, psi		
	75°F	-423°F	600°F
Ultimate, F_{tu}	76,000	240,000	61,000
Yield, F_{ty}	30,000	80,000	23,000

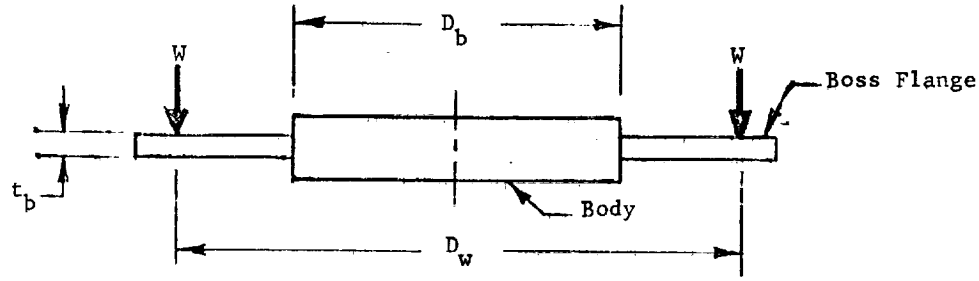
C. DESIGN CRITERIA

The metal boss is to be capable of sustaining the design burst pressure of the stainless steel-lined filament-wound vessel at 75, -423, and 600°F service temperatures. The design burst pressures (p_b) is 3100 psi at both 75 and 600°F, and 4340 psi at -423°F*.

* Based on an anticipated 40% increase in fiber strength at the liquid hydrogen test temperature.

D. ANALYSIS

The critical design condition for the metal boss is the 600°F burst condition. Only the most critical section of the boss, located at the base of the flange, was analyzed. Stresses there were determined by using the conservative assumption that the flange is a flat plate with a concentrated annular load and a fixed inner edge (the body).



The end-for-end wrap pattern of the longitudinal filaments produces a rigid band around the boss that supports the flange. The load applied (W) is the reaction of the boss flange bearing against the composite structure. The total load is therefore equivalent to the pressure acting over the area within the reaction circle. The diameter at which the load is assumed to act (D_w) is (from Reference 3).

$$D_w = (1 + \epsilon_{f,1})\, D_b + 2.0\, W_1$$

where

$\epsilon_{f,1} = \frac{\sigma_{f,1}}{E_f}$ = longitudinal filament strain at failure, in./in.

$\sigma_{f,1}$ = longitudinal filament stress at failure, psi = 287,500 psi

E_f = filament modulus, psi = 12.4×10^6 psi

W_1 = filament-winding tape width = 0.104 in.

D_b = boss diameter = 0.840 in.

The bending stress at the juncture of the flange and boss (σ_b) is calculated in accordance with formulas for loading on a flat plate (Reference 5, Case 22, p. 201):

$$\sigma_b = \frac{\beta_{22} W}{t_b^2}$$

$$W = \frac{\pi p_b D_w^2}{4}$$

$$\beta_{22} \approx \frac{D_w}{D_b} - 1$$

t_b = flange thickness, = 0.125 in.

Solving the relationships

$$\epsilon_{f,1} = \frac{287,500}{12.4 \times 10^6} = 0.0232 \text{ in./in.}$$

$$D_w = (1 + 0.0232)\ (0.840) + 2.0\ (0.104)$$

$$= 1.067 \text{ in.}$$

$$\beta_{22} = \frac{1.067}{0.840} - 1 = 0.27$$

$$W = \frac{\pi\ (3100)\ (1.067)^2}{4} = 2770 \text{ lb.}$$

The bending stress is

$$\sigma_b = \frac{0.27\ (2770)}{(0.125)^2} = 47,900 \text{ psi}$$

and, the margin of safety is

$$\text{M.S.} = \frac{F_{tu}}{\sigma_b} - 1 = \frac{61,000}{47,900} - 1 = \underline{\underline{+\ 0.27}}$$

REFERENCES

1 F. J. Darms, R. Molho, and B. E. Chester, Improved Filament-Wound Construction for Cylindrical Pressure Vessels, ML-TDR-64-63, Volumes I and II, March 1964.

2 F. J. Darms and E. E. Morris, "Design Concepts and Procedures for Filament-Wound Composite Pressure Vessels," Paper presented at American Society for Mechanical Engineers Aviation and Space Conference, 16-18 March 1965, at Los Angeles, California.

3 Structural Materials Handbook, Aerojet-General Corporation, Structural Materials Division, February 1964.

4 F. J. Darms and R. E. Landes, Computer Program for the Analysis of Filament-Reinforced Metal Shell Pressure Vessels, NASA CR-72124 (Aerojet-General Report prepared under Contract NAS 3-6292), May 1966.

5 R. J. Roark, Formulas for Stress and Strain, 4th Edition, McGraw-Hill Book Company, 1965.

TABLE 1

DESIGN CRITERIA

4-in.-dia by 6-in.-long 321 SS-Lined

GLASS FILAMENT-WOUND PRESSURE VESSELS

Geometry and Loading

Diameter, in.	3.942
Length, in.	5.640
Polar Boss Diameter, in.	0.840
Metal Liner Thickness, in.	0.006
Design Burst Pressure at 75°F, psig*	3100.
Winding Tension, lbs/12-end**	6.0

Material Properties

	Type 321 SS Annealed	Glass-Filament-Wound Composite
Density, $lb/in.^3$	0.289	0.072
Coefficient of thermal expansion, in./in. - °F at +75 to +600°F	9.50×10^{-6}	2.010×10^{-6}
Tensile-yield strength, psi	38,000	-
Derivative of yield strength with respect to temperature, psi/°F	-13.0	-
Elastic modulus, psi	28.0×10^6	12.4×10^6 ***
Derivative of elastic modulus with respect to temperature, psi/°F	-8030	-2410
Plastic modulus, psi	84,000	-
Derivative of plastic modulus with respect to temperature, psi/°F	-0.1	-
Poisson's ratio	0.295	-
Derivative of Poisson's ratio with respect to temperature, 1/°F	0.0	-
Volume fraction of filament in composite	-	0.67 **
Hoop filament, design allowable stress at 75°F, psi	-	390,000

* Determined from analysis of other design factors

** Preliminary values

*** Filament Modulus

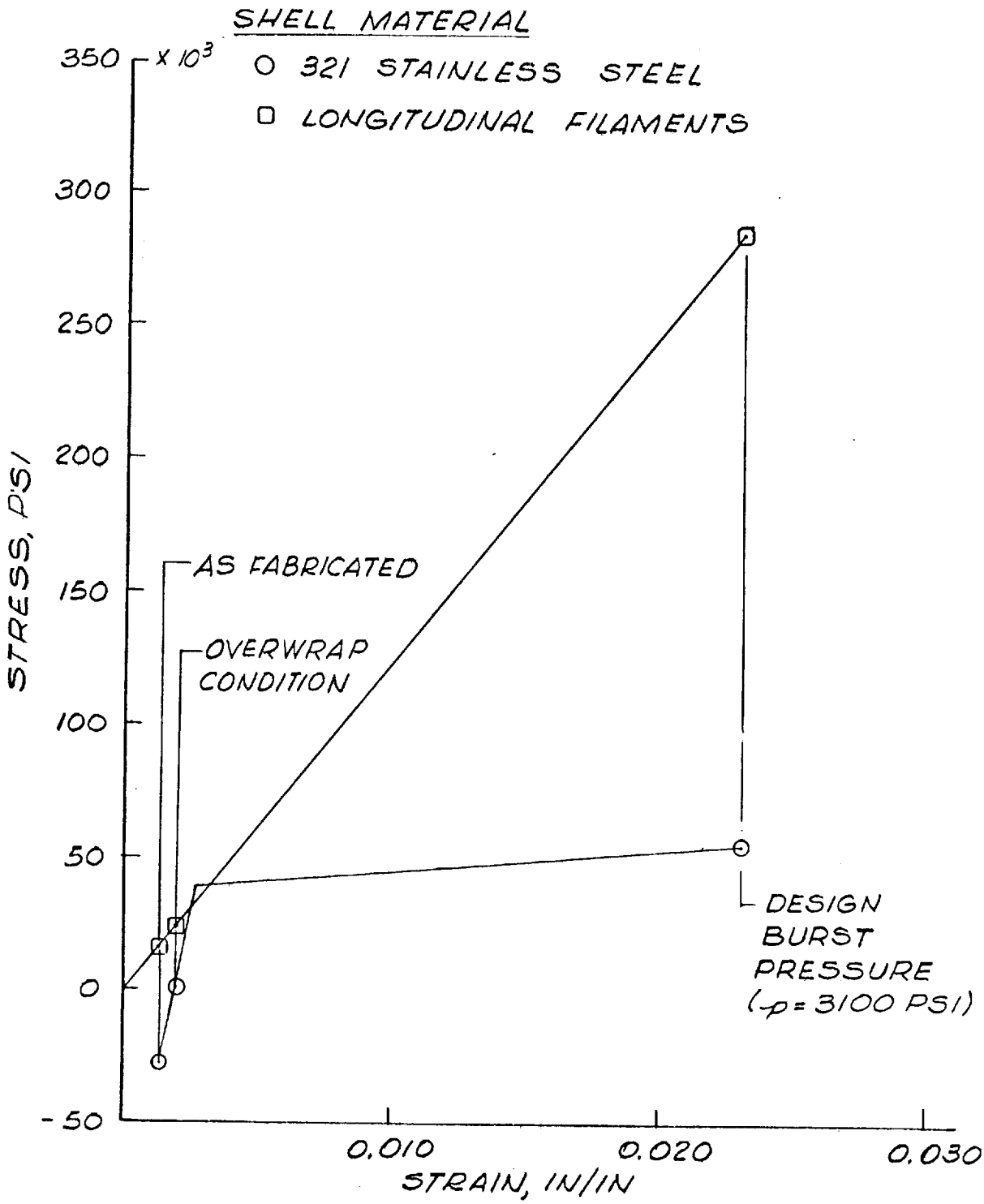


FIGURE 1
AMBIENT STRESS-STRAIN RELATIONSHIPS
LONGITUDINAL DIRECTION OF CYLINDER

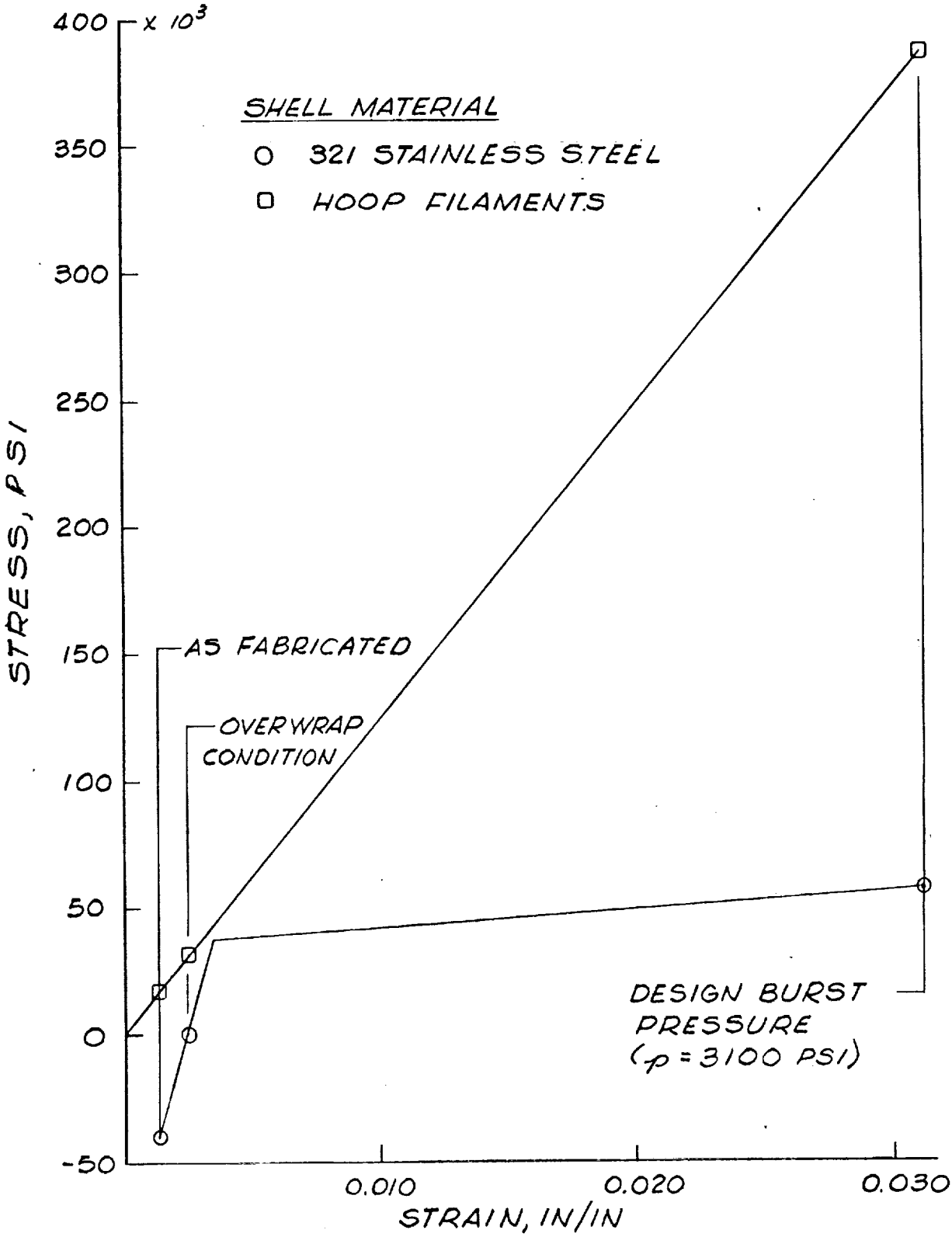


FIGURE 2
AMBIENT STRESS-STRAIN RELATIONSHIPS
HOOP DIRECTION OF CYLINDER

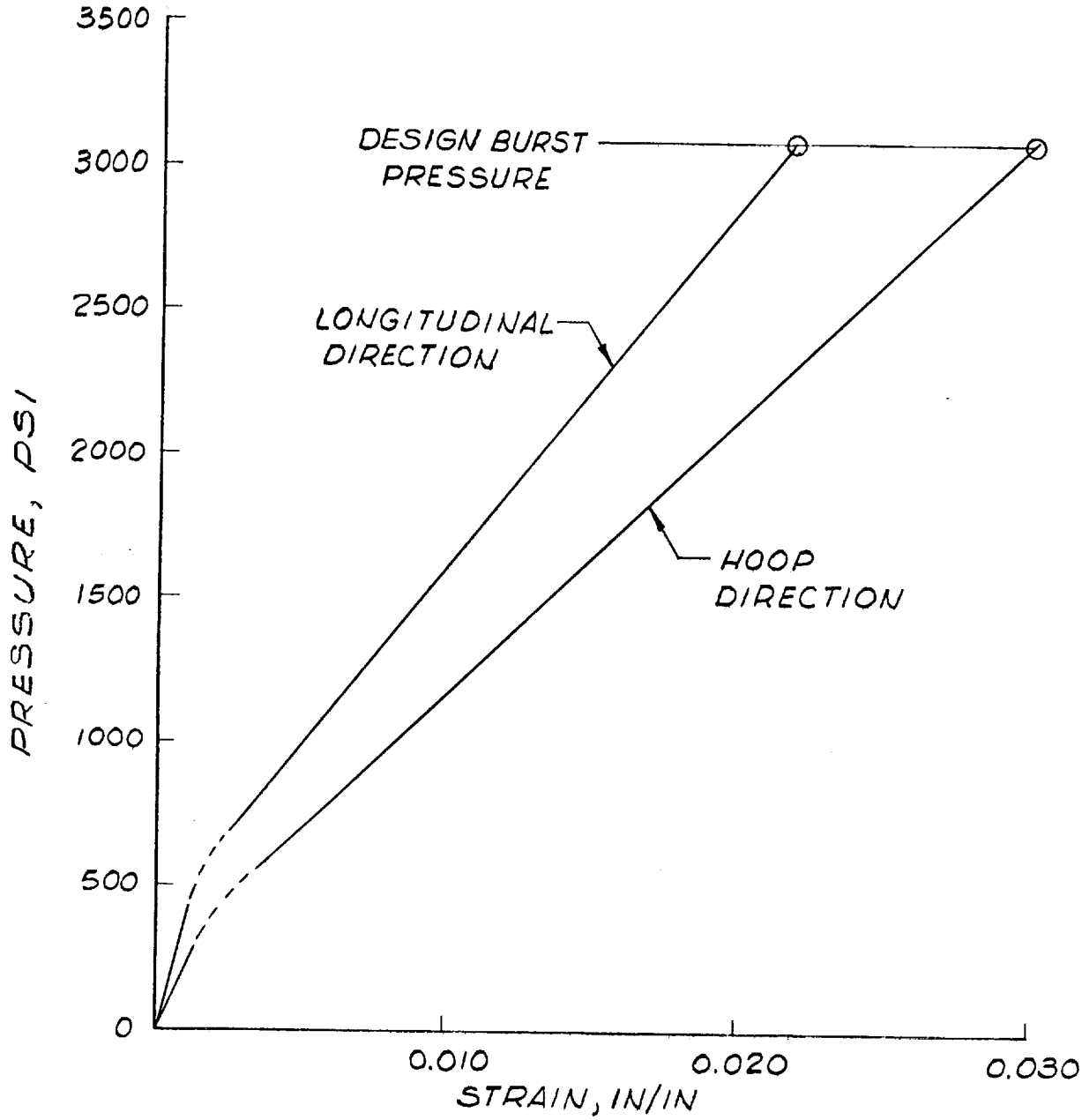


FIGURE 3

AMBIENT PRESSURE - STRAIN RELATIONSHIPS
CYLINDRICAL SECTION PRESSURE VESSEL

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APPENDIX B

LINER ASSEMBLY, PRESSURE VESSEL

STRUCTURAL COMPOSITES INDUSTRIES INC.

6344 NORTH IRWINDALE AVENUE AZUSA, CALIFORNIA 91702 (213) 334-8221

LINER ASSEMBLY, PRESSURE VESSEL

4.00-in.-Dia. by 6.5-in.-long

TYPE 321 STAINLESS STEEL

SPECIFICATION NO. 9141-5

July 1971

Approved By:

Edgar E Morris

E. E. Morris, Vice President
Structural Composites Industries, Inc.

1. SCOPE

1.1 Scope - This specification establishes the requirements for the fabrication and quality conformance inspection of a stainless steel, CRES type 321, liner assembly for use in a glass filament-wound pressure vessel.

2. APPLICABLE DOCUMENTS

2.1 Department of Defense documents - Unless otherwise specified, the following documents, listed in the issue of the Department of Defense Index of Specifications and Standards in effect on the date of invitation for bids, shall form a part of this specification to the extent specified herein.

SPECIFICATIONS

Federal

QQ-S-763B,	Class 321 Stainless Steel, Bars, Forgings Mechanical Tubing and Rings

Military

MIL-T-8606	Type 1, Group 321 Tubing, Seamless
MIL-H-6875D	Heat Treatment of Steel
MIL-I-6866	Inspection, Penetrant Method of

STANDARDS

Military

MIL-STD-453	Inspection, Radiographic

Industry

ASTM-E-8	Tension Testing of Metallic Materials

(Copies of documents required by contractors in connection with specific procurement functions should be obtained as indicated in the Department of Defense Index of Specifications and Standards.)

2.2 Aerojet-General Corporation documents - Unless otherwise specified, the following documents of the latest issue in effect, shall form a part of this specification to the extent specified herein.

SPECIFICATION

AGC-13860	Radiographic Quality Levels, Fusion Weldments

STANDARDS

AGC-STD-7012	Procedure 102, Welding Fusion, Corrosion and Heat Resistant Steels and Alloys
ASD 5215	Marking, Methods of

3. REQUIREMENTS

3.1 Materials

3.1.1 Liner - The liner shell P/N 1269288-2, shall be fabricated from seamless tubing, corrosion resistant steel, Type 321, in accordance with military specification MIL-T-8606, Type 1, Group 321, as the starting material.

3.1.2 Boss - The boss, P/N 1269288-3 shall be fabricated from bar stock, corrosion resistant steel, Type 321, in accordance with federal specification QQ-S-763, Class 321, condition annealled.

3.1.3 Liner Material Identification - Each tube section used for fabrication of P/N 1269288-2, liner shell, shall be identified with a serial number the identification of which shall be maintained during all fabrication operations. This serial number along with the mill heat number for the tube from which the tube section was cut shall be recorded for future reference.

3.2 Design - The metal liner furnished under this specification shall be fabricated in accordance with the requirements of SCI drawing 1269288

3.3 Fabrication of Liner Shell - Unless otherwise specified, liner shell, P/N 1269288-2, shall be fabricated by utilizing the following sequence of operations. The details of each operation shall be the responsibility of the fabricator. Throughout the processing operations care shall be taken to prevent physical damage or chemical contamination to the liner shell.

Operation 1: Cut at least (4) tensile specimens for each mill heat involved and verify satisfactory elongation and tensile strength in accordance with 4.4.2 and 3.6 before forming liner shells.

Operation 2: Prepare tube section for forming. Machine to proper length and break sharp corners inside and out. Degrease and etch clean to remove mill markings and surface oxidation. Serialize with steel stamp within .50 inches of one end, in an area which will not become part of the final liner assembly.

Operation 3: Form liner shell to required shape and size.

Operation 4: Anneal liner shell to remove work hardening as required during forming and upon completion of forming to restore metal to full annealed condition. Annealing shall be performed per requirements established by MIL-H-6875 D and shall be performed in argon atmosphere or vacuum.

Operation 5: Dye penetrant inspect liner shell to insure that there are no cracks or defects in the net area of the shell.

Operation 6: Machine liner shell to obtain finished diameter dimensions for incorporation of boss, P/N 1269288-3, in two locations per drawing 1269288.

Operation 7: Transfer serialization with indelible marking pen to location in cylinder area of liner shell.

3.4 Fabrication of Boss - Unless otherwise specified, the boss shall be fabricated by utilizing the following sequence of operations. The details of each operation shall be the responsibility of the fabricator.

Operation 1: Saw cut bar stock to length to permit ready machining to finished dimensions.

Operation 2: Machine boss complete per SCI drawing 1269288. Match machine 2.202 inch dia. to fit metal to metal with mating diameter hole incorporated in liner shell. Coordinate identification of boss serial number with mating liner shell end hole matched machined.

Operation 3: Identify boss with same assigned serial number and record mating liner shell with specific hole for which boss is dedicated.

Operation 4: Degrease and dye penetrant inspect boss for evidence of cracks and other defects which would render the part inacceptable for the purpose intended.

Operation 5: Store boss with specific liner shell with which it was match machined to fit and maintain as a set of details preparatory to joining.

3.5 Welding of Boss in Liner Shell - A boss, P/N 1269288-3, previously match machined, shall be welded in place at either end of liner shell, P/N 1269288-2. Welding shall be performed in accordance with the following operations:

Operation 1: Etch clean articles to be welded to remove surface contaminants, such as oxide film, which would affect weld performance. Perform final rinse in deionized water.

Operation 2: Wire brush areas proximate to joint just prior to assembly.

Operation 3: Assemble serialized boss to respective liner shell hole to which it was match machined and weld per gas tungsten arc weld process in accordance with AGC-STD-7012, Procedure 102, using CRES Type 321 filler rod, as required.

Operation 4: Dress weld bead to blend with adjacent surface per applicable drawing.

Operation 5: Perform dye penetrant inspection per MIL-I-6866 using solvent soluble dye. Acceptance shall be per 3.8.1.

Operation 6: Leak test welded liner assembly using air at internal pressure of 5 psi. No leakage permitted.

Operation 7: Radiographic inspect welds per MIL-STD-453. Acceptance shall be per 3.8.2.

3.6 Final Fabrication Procedures

Operation 1: Identify liner assembly with part number and serial number, using same serialization as liner shell. Identify by indelible marking pen.

Operation 2: Perform helium leak test of completed liner assembly in accordance with special test procedure prepared for the assembly. Leakage shall not exceed 1×10^{-5} cc/sec at 5 ± 2 psid.

Operation 3: Steel stamp serialization number of acceptable liner assembly on corrosion resistant steel tag and attach to liner assembly. Remove marking pen identification and all other markings from liner assembly with methyl ethyl ketone solvent.

Operation 4: Degrease and anneal liner assembly in vacuum atmosphere per MIL-H-6875 D.

Operation 5: Etch clean liner assembly as required.

Operation 6: Identify liner assembly per 3.11 with same serialization as attached metal tag. Remove metal tag after identification is completed.

Operation 7: Seal in polyethylene bag and store in protective container for subsequent manufacture.

3.6 Test coupons - Prior to starting processing of liner shell P/N 1269288-3, the ultimate tensile strength and elongation in a 2-inch gage length shall be verified with at least 4 tensile specimens from each mill heat involved. The material used for these coupons shall be from the same mill heats used to fabricate the liner shells. Tensile specimens shall be tested in accordance with 4.4.2 and shall comply with the following tensile properties:

(a)	Yield tensile strength, psi	30,000 psi minimum
(b)	Elongation in 2 inches, percent	40% minimum

3.7 Weld repairs - Weld repairs shall be limited to those directed by the project engineer.

3.8 Weld acceptance criteria - Welds shall meet the following quality requirements:

3.8.1 Dye penetrant inspection - The welds shall be free of external cracks or propagating defects. Surface porosity in excess of the limits specified in 3.8.2 is unacceptable.

3.8.2 Radiographic inspection - The welds shall meet the quality level requirements of Specification AGC-13860, Class 11 with the following modifications:

(a) Under scattered porosity - delete 0.010 inch maximum diameter of cavity.

(b) Under excess crown limits substitute "weld crown shall be blended to be smooth with adjacent surfaces."

3.9 Handling - All handling operations of the liner assembly or the liner shells in the uncrated condition shall be performed using maximum care because of the susceptibility of the material to damage during the stages of fabrication. Components or assemblies damaged from handling shall be subject to rejection. The components shall be kept in suitable containers except when they are being worked.

3.10 Cleanliness - After final machining, a cleaning method shall be employed to guarantee the liner assembly interior is completely free from machining residue, shavings, and cuttings. After cleaning, the assembly openings shall remain sealed at all times, except when removal of seals is necessary for final fabrication or testing. The cleaning method shall not damage the materials.

3.11 Identification of liner assembly - Each liner assembly shall be marked with the part number and assigned serial number, by electrolytic etch, as specified by Aerojet Standard ASD 5215, Method C, in the location indicated on Aerojet Drawing 1269288.

3.12 Workmanship - The liner assembly shall be fabricated, annealed, finished, and tested in a thoroughly workmanlike manner. Particular attention shall be given to neatness and thoroughness of the processing and welding of the component parts. Nonconformance to the drawings and the requirements of this specification shall be cause for rejection.

4. QUALITY ASSURANCE PROVISIONS

4.1 Supplier responsibility -

4.1.1 Inspection - Unless otherwise specified, the supplier shall be responsible for the performance of all inspection requirements specified herein and may use any facilities acceptable to the Aerojet-General Corporation (AGC).

4.1.2 Processing changes - The supplier shall make no changes in processing techniques or other factors affecting the quality or performance of the product without prior written approval of SCI.

4.2 Sampling -

4.2.1 Production sample - All production units of the liner assembly, P/N 1269288-1 shall be subjected to quality conformance inspection.

4.3 Quality conformance inspections - Inspection of all liner assemblies shall consist of the following quality conformance inspection to determine compliance with the requirements herein:

(a) Dimensional and visual inspection (see 4.4.1).

(b) Dye penetrant inspection of boss welds (see 4.4.3)

(c) Radiographic inspection of boss welds (see 4.4.4)

4.4 Test methods -

4.4.1 Examination - Each liner assembly shall be measured and visually inspected for conformance to the requirements of Section 3, Section 5 and the drawings.

4.4.2 Tensile strength test method - The tensile strength properties shall be determined in accordance with ASTM-E-8, using the standard sheet-type test specimen with a 2 in. gage length to verify compliance with 3.6.

4.4.3 Dye penetrant inspection - Dye penetrant inspection in accordance with Specification MIL-I-6866, Type I, Method A shall be performed on all welds of each liner assembly to verify compliance with 3.8.1. After inspection the weld shall be thoroughly cleaned.

4.4.4 Radiographic inspection - Radiographic inspection in accordance with MIL-STD-453 shall be performed on all girth welds. Radiographs shall be subject to the interpretation and acceptance in accordance with 3.8.2 by designated Aerojet-General quality control and project representatives. Radiographic film shall be numbered to coincide with the identification markings of the liner assembly. China marking lead shall be used for marking weld identification so that exact locatio of weld areas with corresponding radiographs may be readily identified. All radiographic film shall become the property of the SCI.

5. PREPARATION FOR DELIVERY

5.1 Packing - Liner components, and the liner assembly shall be boxed in a wooden container and firmly supported to prevent damage during storage, handling or shipment.

5.2 Marking - The shipping container shall be marked with the following information:

(a) Manufacturer's name

(b) Part number

(c) Serial number

(d) Number, revision letter, and date of this specification

(e) Purchase order number

6. NOTES

6.1 Intended use - The liner assembly is intended for use as a metal liner for glass filament-wound pressure vessels.

6.2 Ordering data - Procurement documents should specify, but not be limited to, the following information:

(a) Number, revision letter, and date of this specification

(b) Request for three copies of material certification and test results

(c) Responsibility for testing tensile test coupons before forming half-shells (see 3.6)

(d) Operations to be performed by the supplier

(e) Place of delivery for tensile test coupons, half-shell liners, and finished units.

(f) Serial numbers to be assigned.

STRUCTURAL COMPOSITES INDUSTRIES INC.
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APPENDIX C

HELIUM LEAK TEST PROCEDURE

CONTRACT NAS 3-15551

HELIUM LEAK TEST PROCEDURE

FOR

LINER ASSEMBLY, PRESSURE VESSEL

4.00-in.-Dia. by 6.5-in.-Long

TYPE 321 STAINLESS STEEL

SCI DRAWING NO. 1269288

Prepared by S Sillmans 7/17/71

Approved by

HELIUM LEAK TEST PROCEDURE

LINER ASSEMBLY, PRESSURE VESSEL

4.00-in.-Dia. by 6.5-in.-long

TYPE 321 STAINLESS STEEL

I. OBJECT - This test is conducted to verify the structural validity and weld integrity of the stainless steel liner.

II. REQUIREMENTS - Each stainless steel liner shall withstand a 5 ± 2 psig internal proof pressure and the liner shall not show evidence leakage greater than 1×10^{-5} std. cc/sec of helium when subjected to a helium mass spectrometer leak test.

III. TEST SETUP - The setup shall consist of a vacuum chamber surrounding the specimen and a mass spectrometer to monitor the leakage rates. A typical pressure schematic is illustrated in Figure 1.

IV. SAFETY REQUIREMENTS

A. Safety Equipment - Safety glasses will be worn by the personnel during mechanical work in the test bay or when entering the bay for direct test observation.

B. Pressure Restriction - The test bay may not be entered when the specimen pressure exceeds 10 psig.

V. PROCEDURE

A. Carefully remove the liner assembly from the transportation container and inspect for damage. On the Data Sheet, document nicks, scratches, and dents visible on the liner assembly.

B. Verify that all measuring and recording devices are within the certified calibration due date and record measuring equipment in Equipment Log Sheet.

C. Leak Test

1) Setup the liner assembly as shown in Figure 1.

2) Pressurize the liner to 5 ± 2 psig with nitrogen, hold for two minutes and vent to zero. Visually observe the liner during the performance of the proof test for evidence of distortion.

3) Place the vacuum tank over the liner and slowly actuate both the liner and the vacuum chamber by opening vacuum valve #1 and #2 and assuring that #3 valve is closed. Establish a background reference with the Mass Spectrometer.

4) Close vacuum valve #2, open valve #3, and pressurize the liner with helium gas to 5 ± 2 psia.

5) Record the leakage as indicated by the helium Mass Spectrometer leak detector. An acceptable leakage rate shall not be greater than 1×10^{-5} std cc/sec.

6) Enter results of liner leak test in the Data Sheet.

HELIUM LEAK TEST PROCEDURE

DATA SHEET

Test: Helium Leak Test

Date: _____

Start: _____

LINER

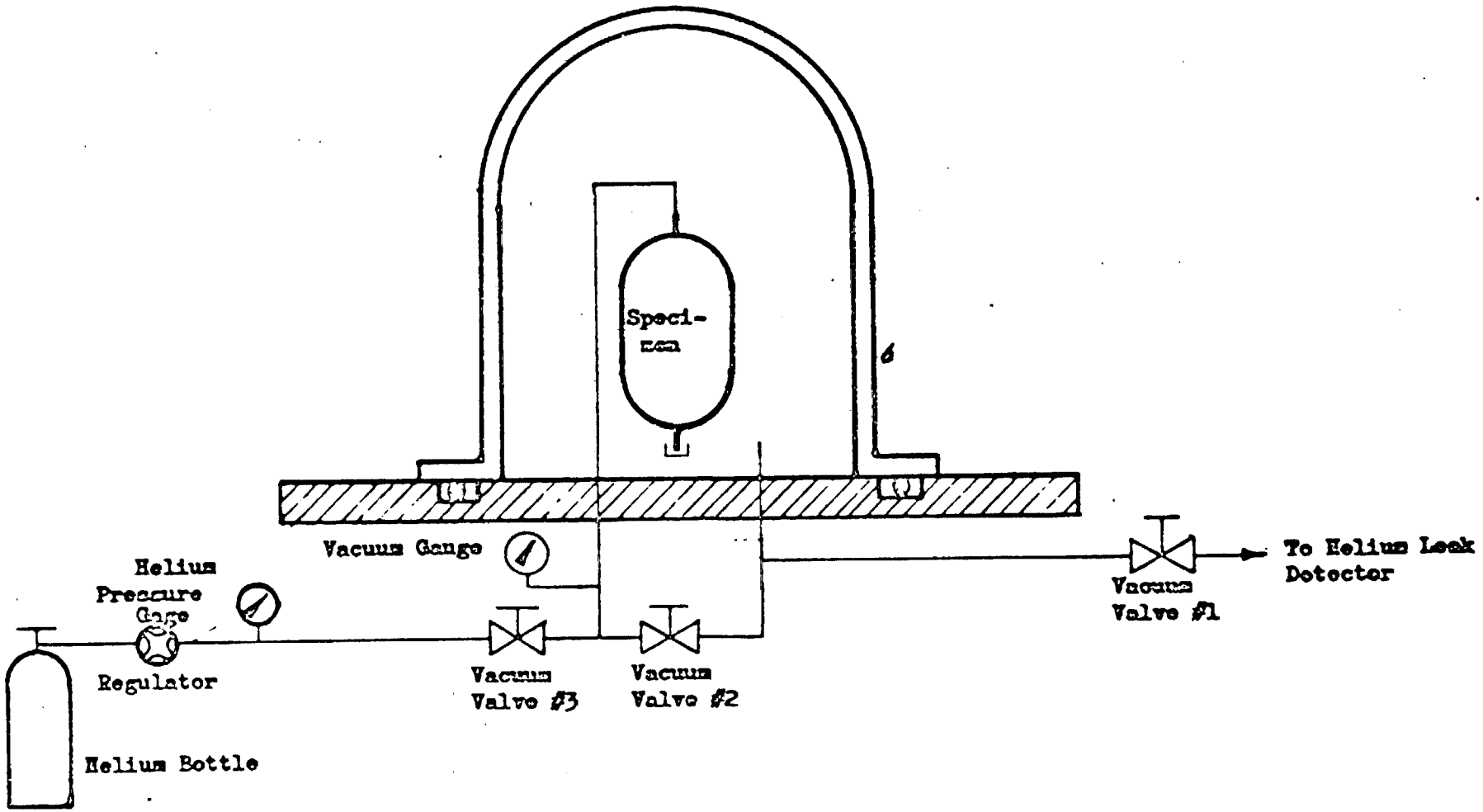
S/N	Proof (psig)	Leakage	Comments

Operator _____

Test Engineer _____

FIGURE I

HELIUM LEAK TEST SCHEMATIC



TEST EQUIPMENT LOG

TYPE OF TEST: _____ PART NO. _____

TEST NO. _____ SERIAL NO. _____

TEST DATE: _____

WORK ORDER: _____

CHECKED BY: _____

DESCRIPTION (MFG. & MODEL NO.)	IDENT. NO.	RANGE	CALIB. DATE

STRUCTURAL COMPOSITES INDUSTRIES INC.

6344 NORTH IRWINDALE AVENUE AZUSA, CALIFORNIA 91702 (213) 334-8221

APPENDIX D

FABRICATION PROCEDURE

FABRICATION PROCEDURE

for

4-In. - Dia by 6-In. - Long

Type 321 Stainless Steel Lined,

Glass Filament-Wound Pressure Vessel

Part No. 1269289

Contract NAS 3-15551
NASA Lewis Research Center
November 1971

Approved by:

Ira Petker, Program Manager

Approved for distribution:

Robert Gordon, President

I. SCOPE

A. This document describes the procedures for fabricating 4-in.-dia by 6-in.-long Type 321 stainless-steel-lined, bidirectional glass filament-wound, pressure-vessel test specimens.

B. This document includes mandrel casting procedure, surface preparation of the metal liner, filament winding, cure procedures, mandrel removal, and liner-vessel final inspection.

C. The processing conditions for filament winding and cure procedures described herein are directed primarily with the use of Gemon L prepreg material.

D. The pressure vessel test specimen is designed for use in determining single-cycle burst strength of the composite material at test temperatures of -423° to 600°F.

II. REFERENCES

A. Drawing 1269288, Liner Assembly

B. Drawing 1269289, Pressure Vessel Assembly

C. SCI Specification No. 9141-5, Liner Assembly, Pressure Vessel 4.0-in.-dia by 6.0-in.-long Type 321 Stainless Steel

III. GENERAL INSTRUCTIONS

A. The Vessel Fabrication Data Sheet is to be filled out in its entirety. Be sure that all weights, dimensions, winding pattern information, cure records, dates, and notes are entered as requested.

B. All weights are to be recorded to nearest 0.1 gram and all dimensions to 0.010 in. or better.

C. It is important that any deviations from the specifications for fabrication be noted on the record in order that all factors may be taken into account when analysis of the vessel is made after test.

D. Verification of calibration status for all data acquisition instruments is required. Serial numbers and calibration dates of instruments are to be recorded, as indicated.

IV. FABRICATION PROCEDURE

A. Casting of Liner Plaster Mandrel

1. Inspect liner for any large discrepancies prior to start of work. Measure liner length, diameters, and weight and record.

2. Insert a plug in one of the liner bosses and stand on its end in position for pouring plaster.

3. Prepare a funnel on the boss by wrapping a 1-in.-wide adhesive tape around it.

4. Mix 1,700 grams Kerr DMM plaster with 510 grams water. Mix thoroughly with hand for 3 to 5 minutes or until a uniform mixture is obtained.

5. Pour the plaster mixture slowly through the boss opening with a steady narrow stream. Avoid covering the boss opening by pouring too rapidly.

6. Fill the vessel liner cavity completely with plaster. Shake or gently tap the liner to settle the plaster and release any occluded air. Filling operation must be completed within 15 minutes from the time water is added to the plaster.

7. Insert a winding drive shaft in the plaster-filled liner through the boss openings. Remove the shaft as soon as the plaster sets.

8. Dry the plaster in an oven for 16 hours at 200°F and 8 hours at 350°F. Take weight measurements before and after drying to determine the thoroughness of drying.

B. Liner Surface Preparation

1. Clean the surface by solvent wiping using a clean white cotton rag moistened with acetone, MEK, isopropyl alcohol, or toluene. Wipe the same surface repeatedly with fresh pieces of cotton rag to make certain that contaminants have been completely removed. An alternate method of cleaning is to sand the surface lightly with 420 grit sandpaper.

2. Cap both ends of the liner and immerse for 10 to 12 minutes in a solution of Prebond 700, 10 to 12 oz. to 1 gallon of water, heated at 180 to 200°F.

3. Rinse with distilled water or deionized water.

4. Immerse in the following solution maintained at 170 to 190°F for 8 to 10 minutes:

sulfuric acid	13.0 parts/volume
hydrochloric acid	7.5 parts/volume
water	79.5 parts/volume

5. Rinse thoroughly by spraying vigorously with deionized water.

6. Dry at room temperature. Apply the primer as soon as possible.

C. Liner Primer Application

1. Thin primer BR-34 with BR-34 thinner at a ratio of 20 parts primer to 7 parts thinner.

2. Apply primer to part using camel or sable hair brush. Desired primer thickness is one to two mils.

3. Dry in air for 1 hour.

4. Place part in an oven at 220°F for 30 minutes and at 410°F for 45 minutes.

5. Proceed to filament wind the vessel or store in a protective bag until ready to overwrap.

D. Winding Machine Setup and Calibration

1. Set the winding gear trains to give 118 turns of longitudinal winding for one complete revolution of the mandrel and 0.052-in. lead per turn of hoop winding (63 turns per 3.114 inches of payoff carriage travel).

2. Install the prepared mandrel and shaft assembly in the winding machine.

3. Dry run the machine (without paying off roving) and check machine settings to obtain the required winding pattern.

4. Install two rolls of 12-end prepreg roving in the tension devices for longitudinal winding and one roll for hoop winding.

5. Set the mandrel in position for longitudinal winding. Ensure that the prepreg roving passes tangent to the bosses with a maximum permissible distance between the boss and tape edge of 0.030 in., by making a few winding arm transverses. Make adjustments as required to provide the specified longitudinal winding pattern.

6. Calibrate the tension devices to provide a dynamic tension of 8 pounds (or other selected tension) per 12-end prepreg roving. Calibrate the tension devices statically then dynamically.

E. Winding Operation

1. General Notes: Stop overwrapping, remove the winding, and restart the process, if any of the following should occur: (a) filament breakage, (b) loss of roving tension, or (c) winding-pattern gapping.

2. Weigh two spools of prepreg roving and install them on the tension devices. Weigh the prepreg spools after completion of longitudinal and hoop windings including any waste. Record the weights.

3. Place a bank of heat lamps, quartz strip heater, and/or heat gun directed at the mandrel on the winding machine so that the mandrel temperature can be maintained at a desired level during winding.

4. Heat the mandrel to 200 to 230°F prior to start of winding. The amount of heat to be applied on the prepreg and mandrel during winding will be controlled qualitatively based on observation with regard to optimum level of resin flow and material compaction as wound on the mandrel. When formation and fusion of resin beads on the outer surfaces of the winding is observed, which is considered an excessive resin flow, reduce the heating on the material.

5. Thread the two prepregs through the guide rollers and payoff head tangent to the metal liner boss and secure them in place.

6. Proceed to wind 118 turns of two-strand tape in longitudinal orientation. At the conclusion of winding, tie the ends of the rovings by burying the end under two or three turns of winding. This is accomplished by overwrapping a folded roving by two to three turns, passing the cut end through the loop, and pulling the looped strand underneath the overwrap.

7. Select winding speeds so as to heat and maintain the prepreg and mandrel temperature at a level for desired resin flow and material compaction. Adjust both the winding speed and heat output from the heating media to obtain the desired condition.

8. Change the machine setup and the liner in position for hoop winding. Wind three layers of prepreg roving along the 3.114-in. cylindrical section. At the end of each layer allow the carriage travel to dwell for one full turn in order to insure the full

thickness before engaging the feed and reversing the direction of carriage travel.

D. Cure and Postcure

1. After winding the hoop layers move the liner back to the longitudinal winding position. Cover the wound assembly with TX-1040 Teflon-impregnated release fabric or equivalent material and overwrap with two strands of 12-end glass roving using the same winding tension and wrap pattern as with the prepreg winding.

2. Apply two layers of glass-roving hoop overwrap in the cylindrical section as with the prepreg winding.

3. Place the wound vessel upright on a rack with the weight resting on a boss.

4. While in this position, cure the vessel in an air-circulating oven. Increase the oven temperature at a rate of 6 to 8°F per minute at up to 350°F. Cure the winding with the mandrel surface temperature of 350°F for 2 hours.

5. Cool the part to 150°F or below before removing the glass-roving overwrap.

6. Examine the composites and note for any anomalies prior to postcure.

7. Postcure the vessel in an oven as follows:

2 hours at 200°F
2 hours at 300°F
2 hours at 400°F
14 - 18 hours at 500°F
2 hours at 550°F

F. Plaster Mandrel Removal

1. Mount the vessel on a plaster wash-out stand in vertical position.

2. Wash out plaster mandrel completely with a 35% solution acetic acid and water.

3. Inspect the liner interior to ensure complete removal of plaster.

4. Dry vessel interior by flushing with acetone or by baking at 250°F for one hour.

G. Vessel Inspection

Measure the finished weight, length, and diameter of the filament-wound vessel.

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APPENDIX E

INSTRUMENTATION AND TEST PROCEDURES

INSTRUMENTATION AND TEST PROCEDURES

FOR

4-IN.-DIA. BY 6-IN.-LONG

TYPE 321 STAINLESS STEEL LINED

GLASS FILAMENT-WOUND/POLYIMIDE

RESIN COMPOSITE PRESSURE VESSELS

SCI Part No. 126289

Contract NAS 3-15551

NASA Lewis Research Center

February 1972

Approved by:

Ira Pelker, Program Manager

Approved for distribution:

Robert Gordon, President

STRUCTURAL COMPOSITES INDUSTRIES INC.

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I. SCOPE

This document establishes the procedures for instrumentation and testing of 4-in.-dia. by 6-in.-long Type 321 stainless-steel-lined glass filament-wound/polyimide resin composite, pressure-vessel test specimens.

II. REFERENCES

A. SCI Drawing 1269288, Liner Assembly

B. SCI Drawing 1269289, Pressure Vessel Assembly

C. SCI Specification No. 9141-5, Liner Assembly, Pressure Vessel 4.0-in.-dia. by 6.0-in.-long Type 321 Stainless Steel

III. INSTRUMENTATION

A. PRIMARY INSTRUMENTATION

The primary test instrumentation, as a minimum, shall consist of transducers, signal conditioning equipment, and recording equipment for recording of the following parameters:

1. Vessel internal pressure
2. Cylinder hoop strain
3. Cylinder longitudinal strain
4. Vessel mean wall temperature

These data shall be recorded continuously as the vessels are pressurized to burst.

B. EQUIPMENT LOGS

Equipment logs shall be prepared and maintained current as a means of documenting the continuous fabrication, assembly, and test history of the item. Entries shall be complete, self-explanatory, and include, but not be limited to, the following:

1. Date of entry
2. Identity of test or inspection
3. Test environmental conditions
4. Characteristics being investigated
5. Performance parameter measurements

6. Complete identification of instrumentation used including serial number and calibration date.

7. Failure observations

8. Repair record

9. Record of pertinent unusual or questionable occurrences involving the equipment.

10. Identity of individual making entry

11. Fabrication, process, and assembly inspection records.

12. Discrepancies between the item tested and pertinent specification and drawings.

C. DATA ACQUISITION EQUIPMENT CONTROL

Equipment used in the acquisition of data shall be calibrated, evaluated, maintained, and controlled to ensure its accuracy and reliability.

1. Calibration - Data acquisition equipment shall be calibrated at scheduled intervals or prior to and after use. The equipment shall be calibrated against certified standards which are readily traceable to National Bureau Standards.

2. Evaluation - Data acquisition equipment shall be evaluated prior to use to determine its accuracy, stability, and repeatability. The evaluation results shall be documented. The evaluation required is dependent on the type of equipment and its intended use under this contract.

a. Commercial equipment for which sufficient information is available relative to its accuracy, stability, and repeatability need not be evaluated if used according to established practice. However, the equipment shall be calibrated and the results documented.

b. Specially designed equipment shall be evaluated. The equipment shall be checked out prior to actual use by using actual test procedures and conditions to verify the suitability of the equipment and use, adequacy of procedures, ease of operation,

accuracy, stability and repeatability. The results shall be documented.

c. Calibration procedures, records, and evaluation documentation on data acquisition equipment shall be available to the NASA Project Manager for review.

3. Specific Requirements

a. All pressure transducers shall be laboratory calibrated at 90 day intervals or sooner as required. The instrumentation systems shall be resistance shunt calibrated prior to each test.

b. All resistance thermometers shall be point calibrated at the applicable cryogenic and elevated temperatures and the instrumentation system shall be calibrated by resistance insertion techniques prior to each test.

c. All thermocouple thermometer instrumentation systems will be calibrated by voltage insertion techniques.

d. All strain displacement transducers shall be calibrated in place on the vessel prior to test using 0 to 0.50-in. as the full deflection range, for hoop deflection, and 0 to 0.12-in. for longitudinal deflection range, employing 1/16 in.-increments during calibration. Keep calibration data for future reference.

IV. BURST TESTS

The burst test shall be performed by increasing the internal vessel pressure at a rate of approximately 1000 psi/min. until failure occurs.

A. NORMAL AMBIENT CONDITIONS

1. Test Conditions

a. Temperature: $77 \pm 18°F.$

b. Pressure: Standard atmosphere

2. Equipment Required

a. Water pressurization system. Estimated ambient temperature burst pressure is 3100 to 3400 psig. System shall be capable of achieving pressurization rate in vessel of 1000 psi/minute up to 3400 psig.

b. Vessel holding stand which will provide a fixed method of connecting the instrumentation and pressurization system to the vessel under test.

c. Data acquisition systems

3. Test Media: Deionized water.

4. Test Procedure:

a. Refer to Figure 1. Measure distance between outside edges of pins on vessel cylinder (L2) and record. Measure vessel diameter (at L1 location) and record. All dimensions shall be accurate to 0.010-in. or better.

b. Functionally check the pneumatic and water flow system.

c. Install the vessel on the holding stand and connect the inlet line to the water system.

d. Fill the vessel with deionized water and flow through the vessel until all the air has been removed. Close the specimen bleed valve.

e. Install instrumentation on vessel (refer to Figure 1) and calibrate the instrumentation as specified in Paragraph III-C.

f. Pressurize the vessel to 100 psig and check the test system for leakage.

g. Pressurize the pneumatic system to 4250 psig. A positive displacement pump of suitable capacity may be substituted for the gas supply system.

h. Turn on the instrumentation recording systems. Pressurize the vessel at a rate of approximately 1000 psi/min until failure occurs.

i. Identify all records and complete the Data and Observation Log.

B. LIQUID NITROGEN CONDITIONS

1. Test Conditions

a. Temperature: -300°F. or lower

b. Pressure: Standard atmosphere

2. Equipment Required

a. Liquid cryogen pressurization system. Estimated cryogenic burst pressure is 4000 to 4400 psig. System shall be capable of achieving pressurization rate in vessel of 1000 psi/min up to 4400 psig.

b. Vessel holding stand which will provide a fixed method of connecting the instrumentation and pressurization system to the vessel under test.

c. Data acquisition systems

d. Cryostat

3. Test Media: Liquid Nitrogen - AFPID 9135-7

4. Test Procedure:

a. Refer to Figure 1. Measure distance between outside edges of pins on vessel cylinder (L2) and record. Measure vessel diameter (at L1 location) and record. All dimensions shall be accurate to 0.010-in. or better.

b. Functionally check the pressurization system.

c. Install the vessel on the holding stand located inside the test chamber.

d. Install instrumentation on vessel (refer to Figure 1) and calibrate the instrumentation as specified in Paragraph III-C.

e. Initiate the liquid cryogen cool down, by filling the test chamber holding the vessel with liquid nitrogen. Stabilize the vessel at liquid nitrogen temperature by complete submersion.

f. Flow liquid cryogen through the vessel until the specified mean vessel wall temperature is obtained.

g. Slowly increase the vessel pressure to approximately 100 psi and check the test system for leakage. Document all parameters as specified in Paragraph III-C.

h. Pressurize the pneumatic gas supply system to 5000 psig. A positive displacement pump of suitable capacity may be substituted for the gas supply system.

i. Turn on the instrumentation recording systems.

j. Pressurize the vessel at a rate of approximately 1000 psi/min until failure occurs.

k. Identify all records and complete the Data and Observation Log.

C. LIQUID HYDROGEN CONDITIONS

1. Test Conditions

a. Temperature: -400°F. or lower

b. Pressure: Standard atmosphere

2. Equipment Required: Same as liquid nitrogen operation

3. Test Media: Hydrogen per BB-H-886b

4. Test Procedure

Perform the test in accordance with the Test Procedure in Paragraph IV-B, 4, a through k, except the vessel shall be filled with glass marbles or other equivalent filler material to reduce the volume of liquid hydrogen in the vessel.

D. ELEVATED TEMPERATURE CONDITION

1. Test Conditions

a. Temperatures: $300 \pm 15°$, $500 \pm 15°$, and $600 \pm 15°$F.

b. Pressure: Standard atmosphere

2. Equipment Required

a. Oil pressurization system. Estimated elevated temperature burst pressure is 2500 to 3400 psig depending on the test temperature varying from 300° to 600°F. The system shall be capable of achieving pressurization rate in vessel of 1000 psi/min. up to 3400 psig.

b. Vessel holding stand which will provide a fixed method of connecting the instrumentation and pressurization system to the vessel under test.

c. Air-circulating oven capable of maintaining temperatures of 300°, 500°, and 600°F.

d. Data requisition systems.

3. Test Media: High flash point (above 600°F.) silicone oil for all elevated temperature tests.

4. Test Procedure:

a. Refer to Figure 1. Measure distance between outside edges of pins on vessel cylinder (L2) and record. Measure vessel diameter (at L1 location) and record. All dimensions shall be accurate to 0.010-in. or better.

b. Functionally check the pressurization system.

c. Install the vessel on the holding stand located inside the test chamber (air-circulating oven).

d. Install instrumentation on vessel (refer to Figure 1) and calibrate the instrumentation as specified in Paragraph III-C.

e. Initiate the vessel heat up by heating the assembly in an oven at the test temperature. Preheat the pressurization silicone oil to test temperature and fill the test vessel. Stabilize the vessel at the test temperature by continued exposure to heat in an oven until the specified mean vessel wall temperature is obtained.

f. Slowly increase the vessel pressure to approximately 100 psi and check the test system for leakage. Document all parameters as specified in Paragraph III-C.

g. Pressurize the pneumatic gas supply system to 4250 psig. A positive displacement pump of suitable capacity may be substituted for the gas supply system.

h. Turn on the instrumentation recording systems.

i. Pressurize the vessel at a rate of approximately 1000 psi/min until failure occurs.

j. Identify all records and complete the Data and Observation Log.

E. THERMAL CYCLING CONDITIONS

1. Test Conditions

   a. Temperature: $77 \pm 18°F.$

   b. Pressure: Standard atmosphere

2. Vessel Prestress

   a. Condition A: No prestress

   b. Condition B: Prestress vessel at 60% of ultimate (or other selected stress level) based on the ambient condition burst strength in IV-A.

3. Thermal Cycling: -320° to 600°F. to room temperature (quench in water) for 100 cycles.

4. Equipment Required

   a. Ambient pressurization system

   b. Vessel holding stand

   c. Data acquisition system

   d. Cryostat

   e. Air-circulating oven capable of maintaining 600°F. temperature

5. Test Media

   a. Pressurization medium: Deionized water

   b. Cryogent: Liquid Nitrogen - AFPID 9135-7.

6. Prestress Procedure

   a. Install the vessel on the holding stand and connect the inlet line to the water system.

   b. Fill the vessel with deionized water and flow through the vessel until all the air has been removed. Close the specimen bleed valve.

c. Pressurize the vessel to 100 psig and check the system for leakage.

d. Pressurize the vessel at a rate of approximately 1000 psi/min. until 1800 psig pressure (60% stress level or to other selected stress level) has been attained. Hold at this pressure for two minutes and then release the pressure.

7. Thermal Cycling Procedure

a. Cool the vessel down at liquid nitrogen temperature by complete submersion in the liquid cryogen. The vessel temperature is considered sufficiently cool when violent boiling of the liquid nitrogen in contact with the vessel subsides to a normal stabilized level.

b. Subject the cooled vessel to heat within 30 to 60 seconds after removing from the cryogen in an oven at 600° F. until the specified mean wall temperature is obtained. Hold at this temperature for 1 minute and then cool to room temperature by exposing the vessel in ambient conditions for 30 to 60 seconds and quenching in water. Monitor the vessel wall temperature initially with a thermocouple thermometer to establish the heat up cycle.

c. Repeat the thermal cycling operations in steps (a) and (b) above for 100 cycles.

8. Burst Test Procedure

Perform the test in accordance with the Test Procedure in Paragraph IV-A, 4.

F. HEAT AGEING CONDITIONS

1. Test Conditions

a. Temperature: $77 \pm 18°F.$

b. Pressure: Standard atmosphere

2. Equipment Required

a. Ambient pressurization system

b. Vessel holding stand

c. Data acquisitions system

d. Air-circulating oven capable of maintaining 600°F. temperature

3. Test Media: Deionized water

4. Test Procedure

a. Subject the vessel to heat in an air-circulating oven at 600°F. for specified duration.

b. Proceed to perform the test in accordance with the normal ambient condition Test Procedure in Paragraph IV-A, 4.

V. DATA REDUCTION

The data reduction shall consist of reducing raw data and plotting the following parameters for ambient, cryogenic, and elevated temperature tests:

A. TEST DATA

The data, as a minimum, shall include the following:

1. Pressure vs. strain

a. Cylinder hoop strain (1 ea.)

b. Cylinder longitudinal strain (1 ea.)

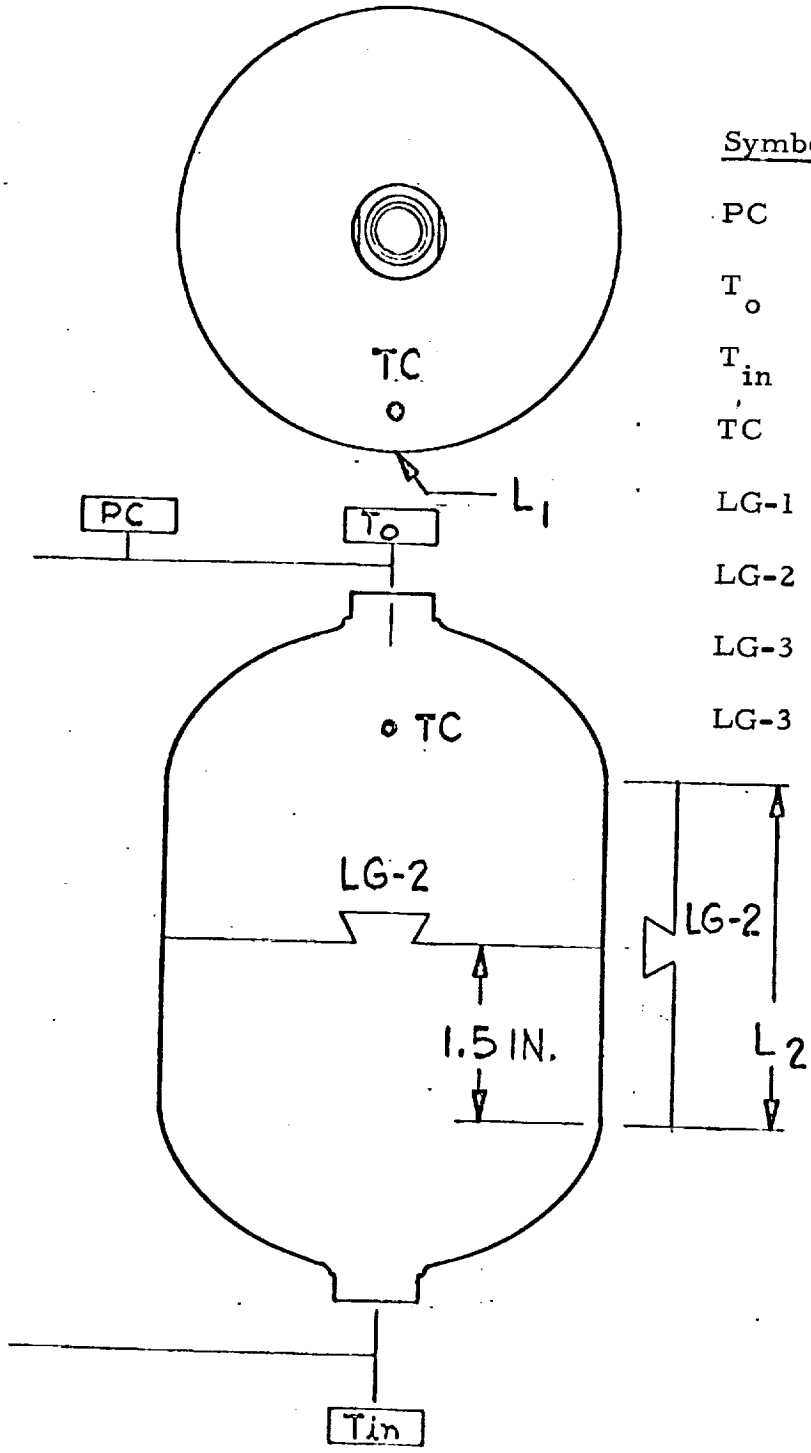
2. Pressure vs. temperature (for cryogenic and elevated temperature tests only)

3. Failure location and mechanisms for each vessel test specimen shall be noted.

B. BURST TEST DATA PLOTS

Plot of vessel test specimen data showing stress vs. strain in both the longitudinal and circumferential windings up to ultimate bursting strength shall be prepared.

C. Ultimate composite and fiber strength as a function of environmental test temperature shall be reported for each vessel test specimen.



Symbol	Measurement
PC	Specimen Pressure
T_o	Specimen Outlet Temperature
T_{in}	Specimen Inlet Temperature
TC	Specimen (Skin) Temperature
LG-1	Specimen Strain, Hoop
LG-2	Specimen Strain, Longitudinal
LG-3	Specimen Strain
LG-3	Strain Measurement Distance

FIGURE 1

6-INCH-DIAMETER PRESSURE VESSEL
INSTRUMENTATION LOCATION